VOL. XXIV 1913

TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY.

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AMERICAN ELECTROCHEMICAL SOCIETY
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TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

VOLUME XXIV

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TWENTY-FOURTH GENERAL MEETING

DENVER, SEPTEMBER 9, 10, 11, 1913

PUBLISHED BY

THE AMERICAN ELECTROCHEMICAL SOCIETY

LEHIGH UNIVERSITY, SOUTH BETHLEHEM, PA.

1914

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VOLUME XXIV. 1913.

TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

PROCEEDINGS

CONDENSED MINUTES OF THE TWENTY-FOURTH GENERAL MEETING OF THE SOCIETY, HELD AT DENVER, BOULDER AND GOLDEN, COLO., SEPTEMBER 9, 10, 11, 1913.

Number of members registered, 52; guests, 59; total, 111.

PROCEEDINGS OF MONDAY, SEPTEMBER 8th.

A considerable body of members and guests attending the meeting arrived in Denver, from the East, at 1.30 P. M. They were met at the station by members of the local committee and friends and were escorted in automobiles about the city, through the parks and boulevards, to the headquarters at the Shirley Hotel.

The Board of Directors met at 8.30 P. M. in the Shirley Hotel.

PROCEEDINGS OF TUESDAY, SEPTEMBER 9th.

The meeting for the reading and discussion of papers was called to order at 10.00 A. M. in the auditorium of the Shirley Hotel, President E. F. Roeber in the Chair.

The Secretary read the following telegram:

> The American Electro-platers' Society wish your Society a pleasant and successful meeting.
>
> F. C. CLEMENT,
> *Supreme Secretary.*

Papers by E. F. Northrup (abstracted by C. A. Hansen), I. Langmuir (presented by C. A. Hansen), J. W. Richards and

E. F. Kern (presented by E. P. Schoch), were presented and discussed as printed in full in these Transactions.

In the afternoon visits were made to the experimental mill and testing plant of Sutton, Steele & Steele Company, the chemical manufacturing plant of the Western Chemical Manufacturing Company, and the general ore testing plant of Henry E. Wood Ore Testing Company.

In the evening visiting members and guests were entertained at a "Smoker" at the University Club.

PROCEEDINGS OF WEDNESDAY, SEPTEMBER 10th.

Special cars were taken on the Denver and Interurban Electric Railway for Boulder. On arrival the buildings of the University were first visited and the visitors were entertained. At 1.00 P. M. lunch was served by the University of Colorado, at the close of which President Roeber expressed the thanks of the Society to its hosts, and President Baker of the University responded in behalf of the University.

A session for the reading and discussion of papers was called to order at 11 A. M. in the Lecture Room of the Physics Laboratory building, at which papers by G. H. Clevenger and M. L. Hall(presented by G. H. Clevenger), and D. A. Lyon and R. M. Keeney (presented by D. A. Lyon) were presented and discussed as printed in full in these Transactions.

As a result of the discussion of Lyon and Keeney's paper, it was moved by Jos. W. Richards and seconded by F. A. Lidbury that it be recommended to the Board of Directors to appoint a committee to confer with the Director of the Bureau of Mines in regard to the activity of said Bureau in electrochemical and electrometallurgical matters. The motion was adopted and at the next meeting of the Board of Directors the following committee was appointed: W. R. Whitney, Chairman; L. Addicks, Wm. Brady, C. F. Burgess, G. H. Clevenger, F. A. Lidbury, and Jos. W. Richards.

At the close of the session the Boulder Motor Club provided automobiles and escorted the members and guests up Boulder Canyon to the power plant of the Central Colorado Power Company, where 20,000 electric horsepower is generated by a 2,500

foot water head. The return to Denver was made late in the afternoon.

PROCEEDINGS OF THURSDAY, SEPTEMBER 11th.

In the morning visits were made in Denver to the testing plant of the American Zinc Ore Separating Company, the lead smelting works of the American Smelting and Refining Company, and the ore dressing plant of the Screenless Sizer Company.

At 11 A. M. special cars were taken for Golden, where lunch was served at noon by the Colorado School of Mines, in Guggenheim Hall. President E. F. Roeber expressed the thanks of the Society to the School of Mines for its hospitality, and President Emeritus Regis Chauvenet responded for the School of Mines.

The session for reading and discussion of papers was called to order at 2 P. M., in the Lecture Room of the Chemical Laboratory. Papers by F. S. MacGregor, S. C. Lind, W. McA. Johnson (presented by Jos. W. Richards), and P. E. Peterson (read by title), A. J. White (read by title), C. W. Bennett and C. O. Brown (read by title), F. C. Mathers (read by title), Alan Leighton (read by title), R. M. Keeney (read by title), Siegfried Fischer (read by title), O. L. Kowalke (read by title), and F. Zimmermann (read by title), were presented and discussed as printed in full in these Transactions.

At the close of the session, a heavy thunderstorm prevented the intended visit to Lookout Mountain and the members and guests were entertained at dinner in Guggenheim Hall by the Colorado Scientific Society. President E. F. Roeber expressed fraternal greetings and thanks to the Colorado Scientific Society, and President Collins of that Society responded. After a very pleasant and sociable evening cars were taken for the return to Denver.

PROCEEDINGS OF FRIDAY, SEPTEMBER 12th.

Train was taken from Denver for Colorado Springs, arriving at 10.30. Street cars were taken for Manitou Springs and then the Cog Wheel Railway to top of Pike's Peak.

In the observatory building on top of Pike's Peak, at 2 P. M., President Roeber called to order the concluding session of the meeting. The Secretary read a petition signed by H. C. Parmelee, of Denver, J. B. Ekeley, of Boulder, and H. J. Wolf, of

Golden, requesting the foundation of a Colorado section of this Society. On motion the request was referred to the Board of Directors with the recommendation that the Section be authorized. The Secretary then offered, and the meeting adopted the following resolution of thanks:

The American Electrochemical Society wishes to record its high appreciation of the assistance of its friends in making possible its very successful meeting in Colorado.

Especially are its thanks due and hereby expressed to the Sutton, Steele & Steele Co., Western Chemical Manufacturing Co., Henry E. Wood Ore Testing Co., Denver Fire Clay Co., American Zinc Ore Separating Co., Screenless Sizer Co., American Smelting & Refining Co., and the Central Colorado Power Co., for the opening of their works to visits and the cordial and painstaking manner in which we were conducted through them; to the Golden Cycle Mining Co., and The Portland Mill for the anticipated pleasure of visiting their mills at Colorado Springs and Cripple Creek. Also to the authorities of the Denver Club and University Club of Denver for their hospitable opening of their club houses to our members and guests. To the Boulder Motor Club for the enjoyable excursion it provided up Boulder Canyon.

To the authorities of the University of Colorado and the Colorado School of Mines, for the facilities provided for our technical sessions and hospitality extended.

To the Colorado Scientific Society for its very fraternal reception and entertainment at Golden.

To the Ladies Committee for the very special attentions paid to the visiting ladies, and to the Local Committee and their numerous friends who worked so industriously and co-operated so effectively in the success of this meeting.

President E. F. Roeber then declared the Twenty-fourth General Meeting of the Society adjourned.

On return to Manitou the Cave of the Winds and the Garden of the Gods were visited by the members and guests.

PROCEEDINGS OF SATURDAY, SEPTEMBER 13th.

At 9 A. M. a party left for a visit to the plant of the Golden Cycle Mining Co., in Colorado Springs, where they inspected this modern mill erected for the roasting and cyanidation of Cripple Creek sulphotelluride ores. Over an hour was spent in the plant under the direction of the Superintendent, Mr. D. Lenoux. Train was taken over the Cripple Creek Short Line to the Cripple Creek district. Detraining near the Portland Mill the party was escorted through this cyanide plant which gave opportunity for observing sand leaching, slime agitation, vacuum filtration and zinc-dust precipitation, the material treated being of gross value less than $2 per ton.

The party returned to Colorado Springs in the late afternoon, and most of the members returned East the same evening.

MEMBERS AND GUESTS REGISTERED AT THE TWENTY-FOURTH GENERAL MEETING.

Members.

T. F. Baily, Alliance, Ohio.
E. A. Beck, New York.
W. W. Case, Jr., Denver, Colo.
Wm. R. Chedsey, Golden, Colo.
G. B. Chorpening, Clarksburg, W. Va.
G. H. Clevenger, Palo Alto, Calif.
Chas. W. Comstock, Denver, Colo.
W. D. Coolidge, Schenectady, N. Y.
Ed. L. Crosby, Detroit, Mich.
W. H. Davis, Boulder, Colo.
D. C. Dodge, Denver, Colo.
Wm. Dreyfus, New York City.
H. C. Eastman, Denver, Colo.
John B. Ekeley, Boulder, Colo.
H. S. Evans, Boulder, Colo.
Colin G. Fink, East Orange, N. J.
Herman Fleck, Golden, Colo.
Francis C. Frary, Minneapolis, Minn.
O. P. Fritchle, Denver, Colo.
Richard H. Gaines, New York City.
A. E. Gibbs, Philadelphia, Pa.
A. McK. Gifford, Pittsfield, Mass.
S. L. Goodale, Pittsburgh, Pa.
J. R. Griffith, Niagara Falls, N. Y.
Henry M. Hall, Massena, N. Y.
C. A. Hansen, Schenectady, N. Y.
A. T. Hinckley, Niagara Falls, N. Y.
Wm. Hoskins, Chicago, Ill.
H. W. Kellogg, Niagara Falls, N. Y.
Geo. I. Kemmerer, Socorro, New Mexico.
F. A. Lidbury, Niagara Falls, N. Y.
S. C. Lind, Denver, Colo.
D. A. Lyon, Pittsburgh, Pa.
F. S. MacGregor, Boston, Mass.
John L. Malm, Denver, Colo.
John A. Mathews, Syracuse, N. Y.
Ralph McNeill, New York City.
L. F. Miller, Golden, Colo.
J. M. Muir, New York.
H. C. Parmelee, Denver, Colo.
J. W. Richards, South Bethlehem, Pa.
E. F. Roeber, New York City.
C. G. Schluederberg, Pittsburgh, Pa.
Herman Schlundt, Columbia, Mo.
E. P. Schoch, Austin, Texas.
G. H. Sethman, Denver, Colo.
Acheson Smith, Niagara Falls, N. Y.
W. M. Snow, Peru, Ill.
Chas. D. Test, Golden, Ohio.
L. D. Vorce, Detroit, Mich.
H. J. Wichmann, Denver, Colo.
H. H. Willard, Ann Arbor, Mich.
Harry J. Wolf, Golden, Colo.

Guests.

John C. Bailar, Golden, Colo.
J. S. Baur, Boulder, Colo.
F. D. Burr, Denver, Colo.
Regis Chauvenet, Denver, Colo.
F. L. Clerc, Boulder, Colo.
C. L. Colburn, Butte, Mont
A. E. G. Collins, Denver, Colo.
Geo. E. Collins, Denver, Colo.
Mrs. George E. Collins, Denver, Colo.
C. L. Devine, Clarksburg, W. Va.
John W. N. Dorr, Denver, Colo.

Guests (Continued).

Mrs. J. W. N. Dorr, Denver, Colo.
Chas. J. Downey, Denver, Colo.
Mrs. H. C. Eastman, Denver, Colo.
E. J. Ericson, St. Louis, Mo.
Mrs. F. C. Frary, Minneapolis, Minn.
A. Galbraith, Chicago, Ill.
F. W. Gill, Arlington, N. J.
Mrs. S. L. Goodale, Pittsburgh, Pa.
R. S. Hawley, Golden, Colo.
Chas. W. Henderson, Denver, Colo.
C. B. Hersey, Denver, Colo.
M. Z. Holt, Denver, Colo.
Arthur J. Hoskin, Chicago, Ill.
Mrs. Hoskin, Chicago, Ill.
J. A. Hunter, Boulder, Colo.
B. H. Jackson, Boulder, Colo.
D. R. Jenkins, Boulder, Colo.
C. S. Johnson, Golden, Colo.
Mrs. Geo. Kemmerer, Socorro, New Mexico.
S. P. Kinney, Salt Lake City, Utah.
Karl L. Kithil, Denver, Colo.
Dr. R. Lachmann, Germany.
F. B. Laney, Washington, D. C.
L. R. Leonard, Boulder, Colo.
O. C. Lester, Boulder, Colo.
H. B. Lowden, Denver, Colo.
N. S. McCarthy, Denver, Colo.
John A. Macdonald, Denver, Colo.
B. S. Manuel, Denver, Colo.
R. B. Moore, Denver, Colo.
J. B. Morrill, Boulder, Colo.
Mrs. H. C. Parmelee, Denver, Colo.
O. C. Ratston, Colo. Springs, Colo.
John W. Root, Denver, Colo.
C. C. Sawders, New York.
Mrs. H. Schlundt, Columbia, Mo.
Mrs. E. P. Schoch, Austin, Tex.
H. M. Showman, Golden, Colo.
Lewis B. Skinner, Denver, Colo.
Mrs. L. B. Skinner, Denver, Colo.
F. W. Skirrow, Westminster, Colo.
H. W. Spicer, Denver, Colo.
F. W. Traphagen, Golden, Colo.
H. V. Welch, San Francisco, Calif.
Edward S. Wiard, Denver, Colo.
M. W. Wilkinson, Denver, Colo.
Miss Constance Wilson, New York.
L. H. Wilson, Newark, N. J.

MEMBERS ELECTED AND QUALIFIED

April, 1913—August, 1913.

April 26, 1913.

W. A. Barnes, Tooele, Utah.
F. C. Woodside, Pittsfield, Mass.
M. W. Merrill, Salisbury, Mass.
W. G. Horsch, Newburyport, Mass.
W. S. Barrows, Toronto, Canada.
N. E. Dabolt, Pittsfield, Mass.
W. O. Boswell, Belleville, Ontario, Canada.
G. A. Perley, Durham, N. H.
G. M. J. MacKay, Schenectady, N. Y.
A. T. Ward, Chrome, N. J.
F. B. Kenrick, Toronto, Canada.
J. E. Breckenridge, Carteret, N. J.
Philip Sievering, Newark, N. J.
DeCourcy B. Browne, New York City.

May 24, 1913.

F. W. Davis, Philadelphia, Pa.
George C. Stone, New York City.
George I. Onions, Buffalo, N. Y.
Warren F. Hubley, East Orange, N. J.
H. G. Lamker, Pittsfield, Mass.
J. M. Lohr, Ithaca, N. Y.
M. L. Hall, Palo Alto, California.

June 27, 1913.

H. C. Ray, Pittsburgh, Pa.
J. W. Offutt, Ellwood City, Pa.
Wm. D. Coolidge, Schenectady, N. Y.
Carl S. Barnes, Niagara Falls, N. Y.
T. M. Caven, Chicago, Ill.
J. B. Glaze, Niagara Falls, N. Y.
Chas. W. Comstock, Denver, Colo.
Verdon O. Cutts, Sheffield, Eng.
Chas. F. Hutchings, Bay City, Mich.

July 25, 1913.

L. J. Renfrew, Dorchester, Mass.
Chas. S. Witherell, Newark, N. J.
Hayes W. Young, Stanford University P. O., California.
H. H. Alexander, Maurer, N. J.
A. R. Ledoux, New York City.
Chas. H. Aldrich, Perth Amboy, N. J.
Herbert I. Allen, Wilmington, Del.
Chas. B. Foley, Dayton, Ohio.
Bhupendranath Ray, San Francisco, Cal.
Harry C. Fernau, Davis, W. Va.

August 22, 1913.

C. C. Titus, Helena, Montana.
Frank E. Barrows, Washington, D. C.
John K. Bryan, Perth Amboy, N. J.
Abbe Sprung, New York City.
L. F. Miller, Golden, Colo.
F. L. Antisell, Perth Amboy, N. J.

AMERICAN ELECTROCHEMICAL SOCIETY.

DIRECTORY OF MEMBERS.

December 1, 1913.

(*Members of date April 3, 1902, are charter members*)

ABBE, Paul O. (Nov. 27, '09) res., 4434 18th Ave., Brooklyn, N. Y.; mailing address, 30 Broad St., New York City.
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ACHESON, Edward G., Jr. (July 31, '08) Old Serjeants Inn Chambers, Chancery Lane, London, W. C., England.
ACKER, Charles E. (Apr. 3, '02) President, The Nitrogen Co., Ossining-on-Hudson, N. Y.
ADAMS, Louis W. (Feb. 25, '11) Assistant Supt., Saucon Plant, Bethlehem Steel Co., South Bethlehem, Pa.; res., 151 S. Center St., Bethlehem, Pa.
ADAMSON, G. P. (Dec. 4, '02) Gen. Mgr., Baker & Adamson Chem. Co.; res., 233 Reeder St., Easton, Pa.
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AIKEN, Robert H. (Oct. 10, '03) Consulting Metallurgist, Winthrop Harbor, Ill.
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AKIZAWA, Rinzo (Jan. 25, '13) Chief Engineer, Nippon Chisso Hiryokaisha, Minamata, Kumamotoken, Japan.
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KENTUCKY.

Danville—
Clarke. F. E.
Marion—
Reed, A. H.

MAINE.

Bangor—
Holman. Chas. Vey
Bath—
Webert, L. P.
Great Works—
Larchar, A. B.
Lewiston—
Robinson, A.
Portland—
Moulton, S. A.
Rumford—
Griffin, Martin L.
Ward, L. E.

MARYLAND.

Annapolis—
McAdam, D. J., Jr.
Baltimore—
Aminoff, G.
Lovelace, B. F.
Murray, W. W.
Peirce, W. H.
Rouse, E. W., Jr.
Wilkinson, T. K.
Luke—
Pedder, John
Sparrows Point—
Martin, S. S.
Weymouth, F. A.
Woodworth, A. J.

MASSACHUSETTS.

Atlantic—
Tomlinson, L. C.
Boston—
Amster, N. L.
Barker, E. R.
Benecke, A. O.
Buchanan, L. B.
Claflin, A. A.
Clapp, E. H.
Eustis, A. H.
Goodwin, H. M.
Little, A. D.
MacGregor, F. S.
Melcher, A. C.
Pratt, F. S.
Sargent, F. C.
Scudder, Heyward
Sharples, S. P.
Skinner, H. J.
Stone, C. A.
Stone, J. S.
Talbot, H. P.
Thompson, M. de K., Jr.
Walker, W. H.
Webster, E. S.
Whiting, J.
Winship, Walter
Brookline—
Howard, H.
Lincoln, E. S.
South Boston—
Ruppel, H. E. K.
Cambridge—
Jones, Grinell
Kennelly, A. E.
Nickerson, W. E.
Richards, T. W.
Walker, C. R.
Yatsevitch, Michael
Dalton—
Newman, M. G.
Great Barrington—
Stanley, Wm.
Lawrence—
Alden, J.
Lynn—
Miller, L. B.
Devers, P. K., Jr.
Malden—
Berry, E. R.
Horsch, Wm. G.
North Woburn—
Mitchell, Thos. A.
Pittsfield—
Dabolt, N. E.
Gifford, A. McK.
Gravely, J. S.
Kelly, J. F.
Lamker, H. G.
Randolph, C. P.
Slade, W. C.
Tressler, M. E.
Unger, M.
Woodside, F. C.
Wooldridge, W. J.
Roxbury—
Werby, A. B.
Salem—
Hamblet, A. M.
Salisbury—
Merrill, M. W.
Southbridge—
Oakden, Wm. E.
Ohlwiler, C. H.
Swampscott—
Thomson, E.

Walpole—
Manahan, Paul R.
West Lynn—
Weintraub, E.
Westwood—
Loring, L.
Wollaston—
Dennison, Chas. H.
Worcester—
Higgins, A. C.
Jeppson, G. N.
Smith, H. B.
Williams, H. J.

MICHIGAN.

Ann Arbor—
Bigelow, S. L.
Cole, Harry N.
Patterson, G. W.
Willard, H. H.
Bay City—
Davies, M. L.
Hutchings, Chas. F.
Detroit—
Crosby, E. L.
Diack, A. W.
Hanks, M. W.
Holland, W. E.
Jenkins, D. J.
Lane, H. M.
MacGregor, Walter
Marsh, A. L.
Shattuck, A. F.
Stephenson, F. T. F.
Stewart, R. S.
East Jordan—
Randall, J. W. H.
Menominee—
White, A. R.
Midland—
Dow, H. H.
Saginaw—
McSweeney, Eugene
Wyandotte—
Vorce, L. D.

MINNESOTA.

Minneapolis—
Frankforter, G. B.
Frary, F. C.
Hunter, J. V.
Lunt, Raymond L.

MISSOURI.

Columbia—
Brown, W. G.
Schlundt, Herman
Kansas City—
Kent, J. M.
Poplar Bluff—
Randall, Merle
St. Louis—
Carrier, C. F., Jr.
Ericson, E. J.
Kohler, H. L.
Queeny, J. F.

MONTANA.

Anaconda—
Eagle, Henry Y.
Butte—
Bowman, C. H.
Lonergan, P. J.
Wells, Roy
Great Falls—
Burns, W. T.
Krejci, Milo W.
Wheeler, A. E.
Wolf, Edgar J.
Helena—
Gerry, M. H., Jr.
Titus, C. C.
Rexford—
Burwell, A. W.

NEBRASKA.

Omaha—
Hall, A. E.

NEVADA.

Goldfield—
Barton, W. H.
Lovelock—
Reid, John T.
Paradise Valley—
Barton, W. B.
Virginia City—
Symmes, W.

NEW HAMPSHIRE.

Berlin—
Barton, C. B.
Perley, G. A.
Keene—
Morgans, F. D.
Wilton—
Abbott, Wm. G., Jr.

NEW JERSEY.

Aldene—
Hayes, G. W.
Arlington—
Joyce, C. M.
Proctor, C. H.
Belleville—
Hungerford, Oliver T.
Bloomfield—
Amer, H. S.
Cowles, H. D.
Frankiln, M. W.
Myers, Ralph E.
Romanelli, E.
Boonton—
Seabury, R. W.
Bridgeton—
Coyle, John A.
Camden—
Benoliel, S. D.
Owen, J. W.
Carteret—
Breckenridge, J. E.
Chrome—
Addicks, L.
East Orange—
Amberg, Richard
Aylsworth, J. W.
Cornell, Sidney
Crumbie, Wm. D.
Elizabeth—
Pyne, F. R.
Ward, A. T.
Gloucester City—
Miner, H. S.
Harrison—
Fink, Colin G.
Hart, L. O.
Kraus, Ernest
Haskell—
Cummings, Wm. J.

Highbridge—
Le Boutillier, C.

Hoboken—
Ganz, A. F.
Stillman, T. B.

Irvington—
Bachofner, D. K.

Jersey City—
Manger, F. W.

Maurer—
Alexander, H. H.
Long, G. E.
Mindeleff, Chas.

Merchantville—
Wickes, C. S.

Montclair—
Crane, F. D.
Pikler, A. H.

Newark—
Colby, E. A.
Fernberger, H. M.
Gifford, W. E.
Heath, H. E.
Heimrod, A. A.
Hogaboom, G. B.
Hubley, W. F.
Liebschutz, M.
Metz, G. P.
Page, Wm. K.
Robinson, F. W.
Sievering, Philip
Skidgell, F. M.
Tessler, A.
Van Winkle, A.
Walsh, P. C., Jr.
Weston, E.
Witherell, C. S.
Zimmermann, F.

New Brunswick—
North, H. B.
Voorhees, L. A.

Orange—
Cunningham, F. W.
Edison, T. A.
McGall, E.
Smith, H. H.

Perth Amboy—
Aldrich, C. H.
Antisell, F. L.
Bryan, John K.
Dill, C.
Emanuel, Louis V.
Fisher H. W.
Foersterling, H.
Gahl, R.
Keller, E.
Nolan, J. J.
Philipp, H.
Renfrew, L. J.
Roessler, F.
Rossi, Louis M.
Sargent, R. N.
Skowronski, S.
Walter, C. A.
Waring, T. D.
Weber, M. G.
Zwingenberger, O. K.

Phillipsburg—
Baker, John T.

Plainfield—
Conlin, F.
Hibbard, H. D.
Van der Linde, Harold
Worth, B. G., Jr.

Princeton—
Hulett, G. A.
McClenahan, H.
Northrup, E. F.

Rahway—
Murray, B. L.

Rutherford—
Daft, Leo
Schroeder, C. M. E.

Sewaren—
Cowles, A. H.
Lemberg, M.

South Orange—
Yunck, J. A.

Trenton—
Stover, Edward C.

Upper Montclair—
Comstock, L. K.
Hemingway, Frank

Watchung—
Moldenke, Richard

NEW MEXICO.

Socorro—
Benjamin, W. W.
Kemmerer, G.

NEW YORK.

Albany—
McElroy, J. F.
Rankin, Herbert E.
Shiverick, Myron D.

Astoria, (L. I.)—
Burby, J. D.

Bedford Hills—
Howe, H. M.

Brooklyn—
Cowan, Wm. A.
Erhart, W. H.
FitzGibbon, R.
Foster, O. R.
Graves, C. A.
Maywald, F. J.
Potthoff, Louis
Sheldon, S.
Sperry, E. A.

Buffalo—
Albright, L.
Bierbaum, C. H.
Childs, D. H.
Corse, Wm. M.
Doty, E. L.
Garretson, Eugene
Green, F. V.
Mantius, Otto
Ramage, A. S.
Skillman, V.
Slocum, Chas. V.
Wilke, Wm.
Zaremba, Edward

Clinton—
Saunders, A. P.

Douglaston, (L. I.)
VomBaur, C. H.

Flushing—
Nash, A. D.

Glen Falls—
Hillard, John D.

Hastings-on-Hudson—
Kenyon, O. A.

Ithaca—
Bancroft, W. D.
Bennett, C. W.
Gillett, H. W.
Lohr, J. M.

LaSalle—
Sneath, Wm. H.

Lockport—
Harris, J. H.
Harrison, H. C.
Howard, L. E.
Kenan, W. R., Jr.

Mamaronech—
Snyder, J. L. K.

Massena—
Hall, H. M.
Herzog, G. K.

Mechanicsville—
Buckie, R. H.

Monticello—
Drobegg, Dr. Gustave
Isakovics, A. von

Mt. Vernon—
Wells, G. A.

New Brighton—
Burger, Alfred

New York City—
Abbé, P. O.
Allyn, R. S.
Aldridge, W. H.
App, O. A.
Baker, H. A.
Barstow, W. S.
Baskerville, C.
Beck, E. A.
Beutner, R.
Bijur, J.
Bjorkstedt, Wm.
Bogue, C. J.
Bowman, W.
Bradley, C. S.
Bradley, W. E. F.
Breneman, A. A.
Brown, H. P.
Browne, D. H.
Browne, deCourcy B.
Buck, H. W.
Buck, L. H.
Caldwell, E.
Cameron, W. S.
Canet, B. Chas.
Carse, D. B.
Case, W. E.
Castle, S. N.
Chandler, C. F.
Clark, W. G.
Clark, W. J.
Cleaves, M. A.
Coho, H. B.
Colcord, F. F.
Colvocoresses, Geo. M.
Corning, C. R.
Crocker, F. B.
Crocker, J. R.
Davis, Chas. H.
Dickinson, W. N.
Doerflinger, W. F.
Doremus, C. A.
Douglas, James
Dreyfus, W.
Du Bois, H.
Duncan, L.
Dwight, Arthur S.
Eimer, A.
Elliott, A. H.
Emerson, H.
Englehard, C.
Eurich, E. F.
Fitz Gerald, C. M.
Fliess, R. A.
Foregger, R. von
Frank, K. G.
Frasch, H. A.
Freas, Thos. B.
Frederick, G. E., Jr.
Fries, H. H.
Gaines, R. H.
Gibbs, W. E.
Goepel, C. P.
Govers, F. X.
Gray, J. H.
Grosvenor, W. M.
Guiterman, K. S.
Halcomb, C. H.
Hansell, N. V.
Harris, J. W.
Hasslacher, J.
Hatzel, J. C.
Heroult, P. L. T.
Hill, N. S., Jr.
Hirsch, Alcan
Hirschland, F. H.
Hoge, J. F. D.
Humbert, Ernest P.
Ingalls, W. R.
Kennedy, J. J.
Kern, E. F.
Kirchoff, C.
Kissock, Alan
Klipstein, E. C.
Kohn, M. M.
Kunz, G. F.
Lamb, A. B.
Langsdorff, J.
Langdon, P. H.
Langton, J.
Leavitt, Wm. F. B
Ledoux, A. R.
Liebmann, A. J.
Lienau. J. H.
Lindberg, S. C: son
Lomax, C. S.
Love, E. G.
Lovejoy, D. R.
Lucke, H. J.
MacDonald, J. A.
Mailloux, C. O.
Martin, T. C.
Marvin, A. B.
Mason, E. J. K.
Mason, F. S.
Mastick, S. C.
McNeill, R.
Mershon, R. D.
Merton, R.
Metz, H. A.
Moody, H. R.
Morgan, J. L. R.
Morrow, J. T.
Morse, W. S.
Muir, J. M.
Muschenheim, F. A.
Nichols, W. H.
Nichols, W. S.
Nodell, W. L.
Ornstein, G.
Parker, H. C.
Pattison, F. A.
Peacock, Samuel
Pearson, F. S.
Pennie, J. C.
Price, E. F.
Prindle, E. J.
Prosser, H. A.
Riglander, M. M.
Riker, J. J.
Robb, C. D.
Roeber, E. F.
Roller, F. W.
Rossi, A. J.
Ruhl, L.
Schloss, J. A.

Schuetz, F. F.
Seward, G. O.
Sharp, C. H.
Slocum, H. J.
Smith, Dyer
Smith, E. A. C.
Speiden, C. C.
Spice, R.
Spilsbury, E. G.
Statham, Noel
Stone, Geo. C.
Stone, I. F.
Stoughton, Bradley
Stutz, E.
Swenarton, W. H.
Takamine, J.
Taylor, J. R.
Teeple, J. E.
Thatcher, C. J.
Thompson, J.
Thompson, R. N.
Toch, M.
Tucker, S. A.
Van Arsdale, G. D.
Waddell, M.
Waldo, W. B.
Walker, A. L.
Waterman, F. N.
Weeks, F. D.
Weinstein, L. J.
White, J. G.
Widmann, E. A.
Wiechmann, F. G.
Wigglesworth, H.
Williams, A.
Williams, J. T.
Wood, E. F.
Wright, Arthur

Niagara Falls—
Allen, T. B.
Arison, W. H.
Barnes, Carl S.
Barton, P. P.
Becket, F. M.
Beckman, J. W.
Bennie, P. McN.
Bliss, Wm. Lord
Brockbank, Clarence J.
Callahan, J. F.
Carveth, H. R.
Cole, E. R.
Converse, V. G.
Corbin, J. Ross
Cox, G. E.
Dunlap, O. E.
Edmands, I. R.
FitzGerald, F. A. J.
Fowler, R. E.
Furness, G. C.
Giles, I. K.
Glascock, B. L.
Glaze, John B.
Griffith, J. R.
Hall, C. M.
Harper, J. L.
Haskell, F. W.
Hepburn, D. M.
Hinchley, A. T.
Horry, W. S.
Hooker, A. H.
Hutchins, Otis
Imlay, L. E.
Johnson, J. A.
Kellogg, H. W.
Kemmer, F. R.
Koethen, F. L.
Lidbury, F. A.
MacMahon, J.
Marsh, C. W.
Marshall, J. G.
Mauran, M.
McCullough, H. F.
Meredith, W. F.
Moritz, C. H.
Moyer, G. C.
Petinot, N.
Phillips, R.
Reeve, A. G.
Ross, Wm.
Russell, C. A.
Saunders, L. E.
Sergeant, E. M.
Simmers, A. L.
Smith, E. S.
Smith, W. A.
Snowdon, R. C.
Speiden, E. C.
Sprague, E. C.
Stamps, F. A.
Stone, G. W.
Taylor, Chas. E.
Tone, F. J.
Vaughn, C. F.
von Kugelgen, Franz
Wallace, W.
White, R. H.
Wilson, J. R.
Williamson, A. M.

Ossining—
Acker, Chas. E.

Penn Yan—
Taylor, E. R.

Piermont—
Main, W.

Poughkeepsie—
Moulton, C. W.
Quintard, Edw.

Prince Bay (S. I.)—
Johnston, F. A.
Johnston, W. A.

Richmond Hill (L. I.)—
Haslwanter, C.
Maeulen, F.
Smull, J. G.

Rochester—
Hutchings, J. T.
Schwarz, R. C.

Schenectady—
Andrews, W. S.
Arsem, W. C.
Arthur, Walter
Berg, E. J.
Capp, J. A.
Coffin, F. P.
Coolidge, Wm. D.
Creighton, E. E.
Dushman, Saul
Hansen, C. A.
Hawkins, L. A.
Lamar, M. O.
Langmuir, I.
MacKay, G. M. J.
Murphy, E. J.
Reist, H. G.
Rushmore, D. B.
Seede, J. A.
Steinmetz, C. P.
Taylor, J. B.
Valentine, I. R.
Van Brunt, C.
Whitney, W. R.
Willey, L. M.

Sea Gate—
Rowand, L. G.

Syracuse—
Brookfield, W. B.
Matthews, J. A.
Pennock, J. D.
Troy—
Hunter, M. A.
Lincoln, A. T.
Utica—
Johnson, M. H.
Watertown—
Williams, F. M.
Yonkers—
Baekeland, L. H.
Harrington, E. I.

NORTH CAROLINA.

Charlotte—
Gilchrist, P. S.
Clemmons—
Pickens, Rufus H.
Stackhouse—
Betts, A. G.
West Raleigh—
Browne, W. H.
Whitney—
Marseilles, Wm. P.
Rockwell, Wm. F.

NORTH DAKOTA.

Agricultural College—
Dolt, Maurice L.

OHIO.

Akron—
Shaw, E. C.
Alliance—
Baily, T. F.
Briggs, F. H.
Cope, F. T.
Tombaugh, S. F.
Canton—
Miller, Wm. H.
Cincinnati—
Aston, James
Nash, C. A.
Cleveland—
Brady, W. B.
Brown, J. W.
Chillas, R. B., Jr.
Clymer, W. R.
Collens, 2d, C. L.
Cook, Edw. B.
Crider, J. S.
Fleming, S. H.
Graves, W. G.
Hamister, V. C. H.
Hill, Chas. W.
Hudson, A. J.
Hyde, E. P.
Koehler, W.
Mann, W. W.
Smith, A. W.
Wellman, S. T.
Whitlock, E. H.
Wilcox, W. G.
Woodward, J. M.
Columbus—
Allensworth, H. R.
Flowers, A. E.
Withrow, J. R.
Dayton—
Clements, F. O.
Deeds, E. A.
Dorsey, H. G.
Foley, Chas. B.
Niswonger, E. E.
Pritz, W. B.
Elyria—
Boynton, A. J.
Fostoria—
Downes, A. C.
Fremont—
Goodwin, J. H.
Ziegler, W. W.
Lakewood—
Mott, W. R.
Middletown—
Ahlbrandt, G. F.
Oberlin—
Crafts, W. N.
Salem—
Davis, D. L.
Steubenville—
Homan, J. G.
Warren—
Richardson, E. A.
Youngstown—
Brinker, H. L.
Danforth, Chas. W.

OREGON.

Eugene—
Shinn, F. L.

PENNSYLVANIA

Altoona—
Casselberry, H.
Hanger, S. R.
Ambler—
Coleman, Wm. B.
Ambridge—
Farnham, F. F.
Ashbourne—
Pickering, O. W.
Aspinwall—
Hessom, B. F., Jr.
Beaver—
Baltzell, W. H.
Cornell, Sidney
Brackenridge—
Ober, J. E.
Braddock—
Friedlaender, E.
Lewis, J. D.
Bridgeville—
Clark, W. W.
Saklatwalla, B. D.
Bryn Mawr—
Ely, T. N.
Canonsburg—
Bleecker, W. F.
Guerber, A. J.
Canton—
Taylor, F. D.
Chester—
Comey, A. M.
Coatesville—
Coates, J.
Coraopolis—
Stafford, S. G.
Creighton—
Gelstharp, F.
Kerr, Chas. H.
Parkinson, J. C.
Crofton—
Fawcett, L. H.
Duquesne—
Unger, J. S.
Easton—
Adamson, G. P.
Gordon, C. McC.
Hart, E.
Shimer, E. B.

Economy—
Farnham, F. F.
Ellwood City—
Barton, Chas. R.
Dunn, J. J.
Offutt, J. W.
Erie—
Diller, H. E.
Rice, Edw. T.
Gettysburg—
Parsons, L. A.
Harrisburg—
Calder, A. R.
Ingram—
Armor, J. C.
Johnstown—
Appel, M.
Kenney, E. F.
Parkhurst, C. W.
Latrobe—
Eckfeldt, J. J.
Lebanon—
Crowell, W. J., Jr.
Weimer, E. A.
McKeesport—
Clarke, E. B.
Garratt, Frank
Goodspeed, G. M.
Hensen, Emil
Moore—
Werliin, G.
Munhall—
Hartzell, L. M.
Hopkins, G. A.
James, E. T.
McNiff, G. P.
Reese, P. P.
New Castle—
Samuels, Wm. P.
New Kensington—
Wilson, I. W.
Oakmont—
Keeney, R. M.
Palmerton—
Holdstein, L. S.
Parnassus—
Blough, Earl
Philadelphia—
Bacon, E. W.
Bonine, Chas. E.
Bower, F. B.
Breed, G.
Cary, C. R.
Chance, H. M.
Clamer, G. H.
Davis, F. W.
Davis, R. W., Jr.
Devereux, W.
Eglin, W. C. L.
Flagg, S. G., Jr.
Fraley, J. C.
Furness, R.
Gerety, J. J.
Gibbs, A. E.
Gille, C. H.
Goldbaum, J. S.
Hering, C.
Hess, H.
Hicks, Edwin F.
Hildebrand, J. H.
Hitchcock, F. R. M.
Hornor, H. A.
Howard, G. M.
Keith, N. S.
Kitsee, I.
Lafore, J. A.
Lavino, E. J.
Lay, J. Tracy
McConnell, J. Y.
McFarlin, J. R.
Merzbacher, Aaron
Meyer, J.
Morgan, Leonard P.
Paul, H. N.
Reed, C. J.
Rosengarten, G. D.
Russell, C. J.
Sadtler, S. P.
Sadtler, S. S.
Salom, P. G.
Schamberg, M.
Smith, E. F.
Smith, E. W.
Snook, H. C.
Steinmetz, J. A.
Stevens, J. F.
Taggart, W. T.
Thwing, C. B.
Weeks, C. A.
Weisenburg, Andrew
Weitlaner, R. J.
White, A. J.
Wirt, Chas.
Pittsburgh—
Babb, Jos. E.
Bennett, B. F.
Bradley, Jas. H.
Brill, Otto
Brown, John T., Jr.
Connell, W. H.
Crabtree, F.
Davis, S. A.
Dewey, B.
Donkin, Wm. A.
Dougherty, J. W.
Flannery, Jas. J.
Gibson, C. B.
Goodale, S. L.
Hartley, R. H.
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A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Denver, Colorado, September 9, 1913, President E. F. Roeber in the Chair.

FLOW OF HEAT THRU FURNACE WALLS: THE SHAPE FACTOR.

By Irving Langmuir, E. Q. Adams and G. S. Meikle.

The ordinary formula for the flow of heat thru plates or rods of solid materials may be written:

$$W = \frac{A}{t} k (T - T_0) \qquad (1)$$

where W = the heat flow, expressed in watts
A = area of plate or cross-section of the rod
t = thickness of the plate or length of the rod
$T - T_0$ = the difference of temperature between the two sides of the plate or between the ends of the rod
k = heat conductivity of the material of the plate or rod in watts per cm. per degree.

In most practical cases of heat flow the problem is more complicated than this. Usually the heat is not flowing between parallel surfaces, or at least the areas of the two bounding surfaces are different. In such cases the cross-section A of the path of the heat may vary widely in different parts of the path, and even the length of the path may vary between different parts of the surfaces. That is, both A and t may be complicated variables, so that the above formula loses its significance.

For the case of the conductance of heat thru furnace walls, Mr. Carl Hering (Trans. Amer. Electrochem. Soc. **14**, 215 (1908)) has clearly pointed out the serious errors that may be made by applying the above formula. He shows that the common practice of taking the average area of the inner and outer surfaces of the furnace walls and substituting this for A in equation (1) may lead to results which are so inaccurate as to be very misleading.

Hering then gives formulas, obtained by E. F. Northrup, for calculating the conductances thru such elementary shapes as

spherical shells, cylindrical shells and cubical shells. For walls of other shapes he shows how, for practical purposes, they may be reduced to one or more of these elementary shapes, and approximate solutions much better than those usually employed may be obtained.

The formulas given by Hering for the spherical and cylindrical shells are rigorous, but the formula for the cubical shell (or frustrum of a pyramid), which he considers "rigidly correct," is only approximate.

Some time ago (August, 1911) one of us had occasion to calculate the heat that would flow out from a rectangular casting thru a uniform layer of insulation covering it. A rigorous solution of the problem is extremely difficult, but it is easy to obtain two methods, one which must give a result too high and the other a result too low, so that the true result is known to lie between them.

The equations thus obtained have the advantage of very great simplicity, and by the experimental determination of a few constants they can be made more accurate than any hitherto given. Since such formulas are of importance in the design of electric heating devices and electric furnaces, it was decided to make the necessary experimental investigation.

The present paper deals with the results of this theoretical and experimental investigation.

Equation (1), as it stands, applies only to the flow of heat between parallel surfaces thru a body of uniform cross-section. For bodies of other shapes, A will vary along the path of heat flow. However, if the area of inflow and outflow are fixt, the ratio A/t will have a definite value, depending only on the shape of the body. This quantity we shall call the shape factor, and will represent it by S. Our formula thus becomes:

$$W = Sk\,(T - T_0) \tag{2}$$

In case the heat conductivity is a function of the temperature, the equation should be written:

$$W = S \int_{T_0}^{T} k\,dT \tag{3}$$

The shape factor has the dimension of length, and hence if it is known for a body of any given shape it may be found for any

geometrically similar body by multiplying by the ratio of corresponding linear dimensions.

I. Theoretical Determination of the Shape Factor.

(1st Case) Parallel Plane Surfaces: In those cases where the isothermal surfaces are a series of parallel planes and where the cross-section of the heat path is constant thruout the length of the path, the shape factor is:

$$S = \frac{A}{t} \tag{4}$$

This formula applies to the conduction thru thin slabs or plates of uniform thickness, and also to the conduction thru rods of uniform cross-section, provided the heat loss from the lateral surface of the rod may be neglected.

(2nd Case) Concentric Cylinders: Let us consider the case of a circular cylinder of length l and diameter a, covered with a uniform layer of insulation, of thickness t. Let b be the diameter of the outside surface of the insulation, so that $b - a = 2t$. Then if the cylinder is so long that the heat loss from the ends may be neglected, it can readily be shown that the shape factor of the insulation is:

$$S = \frac{2\pi l}{\log_e \frac{b}{a}} = \frac{2.73\, l}{\log \frac{b}{a}} \tag{5}$$

For infinitely long cylinders this formula is rigorous.

(3rd Case) Concentric Spheres: Where we have a sphere of diameter a covered with a uniform thickness t of insulation, then it can be readily shown that the shape factor for the insulation is:

$$S = \frac{2\pi}{\frac{1}{a} - \frac{1}{b}} = \frac{2\pi ab}{b - a} = \frac{\pi ab}{t} = \frac{\sqrt{AB}}{t} \tag{6}$$

Here b is the diameter of the sphere outside the insulation; that is, $b = a + 2t$. The surfaces of the inner and outer spheres are represented by A and B.

These formulas also are rigorous.

(4th Case) Rectangular Prisms: Let us consider the case where the inside of a furnace is rectangular and where the walls are built of brick and are of uniform thickness. If the thickness of the walls is very small compared to the dimension of the furnaces, then the area of the inside and outside surfaces of the wall will differ relatively little, so that no great error will be made if the shape factor be taken to be A/t, where A is the average of the inside and outside areas and t is the thickness of the walls.

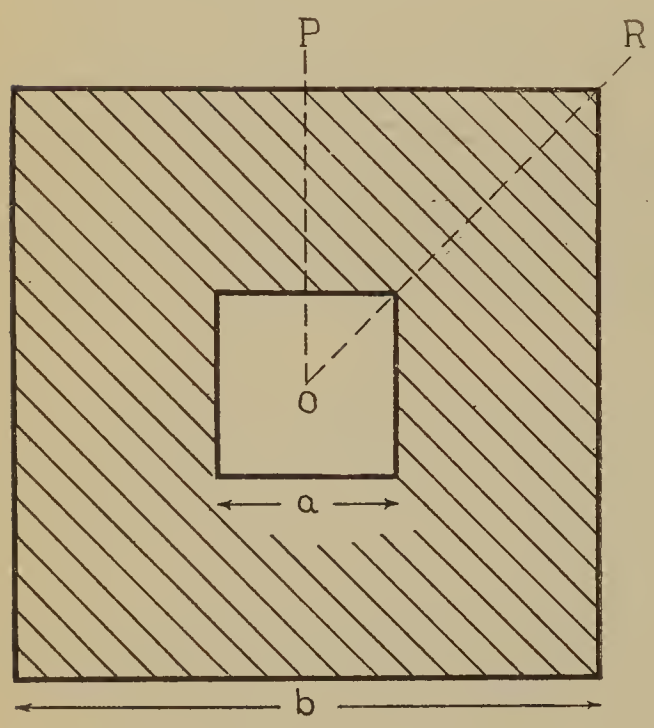

FIG. 1.

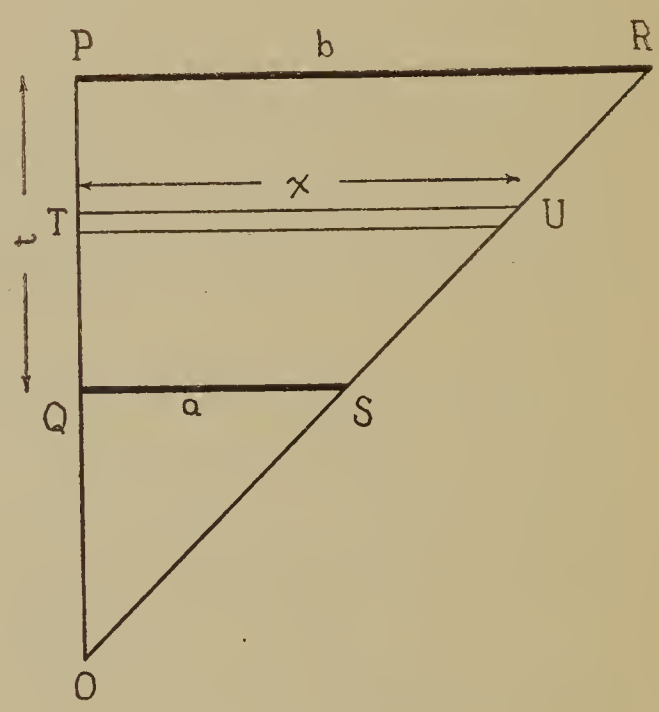

FIG. 2.

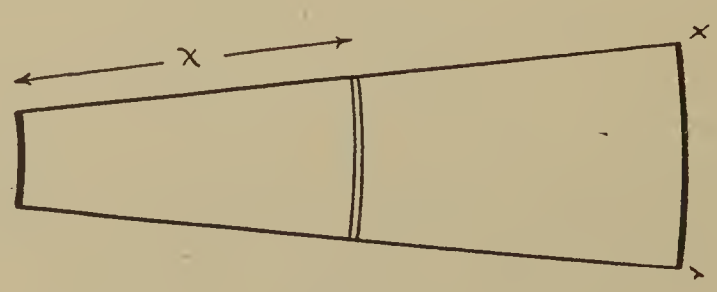

FIG. 3.

Where the thickness of the walls is relatively great, this method (as Hering has shown) leads to serious error.

The simplest case of this kind is that of a furnace in which the hot zone is in the shape of an infinitely long square prism. The cross-section of such a furnace is represented in Fig. 1. We can simplify the handling of this case by considering only that part included between PO and RO. The shape factor for the whole insulation will then be eight times that for this part.

In Fig. 2 POR represents the sector we are considering. The surface QS is part of the inside of the furnace, and the heat is conducted thru the insulation to the surface PR.

First Method. Determination of an upper limit for the shape factor. The method commonly employed in handling a problem of this sort is to consider the cross-section of the heat path to vary according to some function of the distance along the path. For example, if we take the case of heat flowing along a wire of variable diameter, Fig. 3, we would proceed as follows:

Let A be the area of cross-section at a distance x from the end. Then if we apply equation (1) to an element of length, we would have:

$$W = \frac{A}{dx} kdT \tag{7}$$

whence, since W is the same thruout the length of the wire,

$$W \int \frac{dx}{A} = \int kdT \tag{8}$$

or

$$W = \frac{1}{\int \frac{dx}{A}} \int kdT \tag{9}$$

By comparing this with (3), we see that the shape factor S for a rod of variable cross-section is:

$$S = \frac{1}{\int \frac{dx}{A}}. \tag{10}$$

Similarly, it can be shown that the shape factor for a plate of variable thickness is given by:

$$S = \int \frac{dA}{t} \tag{11}$$

Let us now apply this method to the case represented by Figs. 1 and 2. Here the area of cross-section varies from al to bl, where l = the length of the prism, $b = PR = PO$ and $a = QS = QO$. Applying equation (10) to this case, we have

$$S = \frac{1}{\int_a^b \frac{dx}{lx}} = \frac{l}{\log_e \frac{b}{a}} \tag{12}$$

For the whole prism the shape factor would be:

$$S = \frac{8l}{\log_e \frac{b}{a}} \tag{13}$$

It is evident, however, that this formula is only a rough approximation, for in its derivation it has been tacitly assumed that the surfaces TU are isothermal surfaces.

As a matter of fact, the true isothermal surfaces would curve over the edge about as indicated in Fig. 4. The case that we have actually considered could be realized in practice if we could embed in the insulating material a series of infinitely thin but

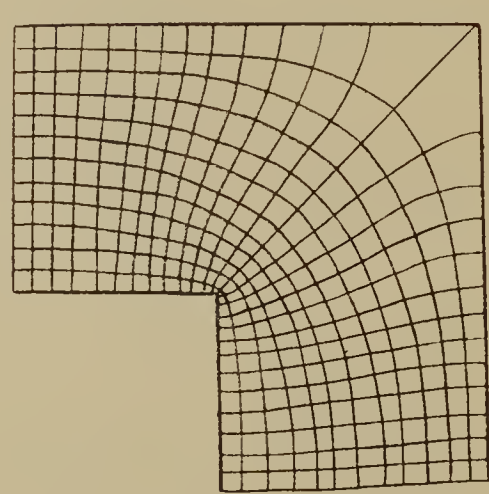

Fig. 4. Flow Lines and Isothermal Lines Near a Square Edge.

perfectly conducting sheets of metal parallel to the surface PR as indicated in Fig. 5. Because of the heat conducted along these sheets, they would become isothermal surfaces. It is evident that any such placing of conducting sheets in an insulating material will tend to lower its thermal resistance, which is equivalent to increasing the shape factor. Therefore, the above method gives us a value for the shape factor which is necessarily too high. Equation (13) gives us, therefore, an upper limit for the shape factor.

If we can now find a method of determining a lower limit for the shape factor, we can estimate the magnitude of the error likely to be made by applying this equation.

In our previous method we made assumptions equivalent to compelling the heat to flow in a way amenable to calculation by interposing conducting surfaces across its path. An analogous

result might be obtained by compelling the heat to flow in the desired manner by confining it between surfaces which we imagine to be perfect heat insulators. The interposition of such surfaces, of course, will lead to resistance higher than the actual ones, equivalent to shape factors less than the true ones.

The actual path of the heat as represented by the flow lines must be everywhere perpendicular to the isothermal surfaces. Such curves are drawn in Fig. 4. It is evident by inspection of this figure that the density of the heat flux must be greatest near the edge of the inner prism, and least near the edge of the outer one. In the previous method of calculation the heat flux

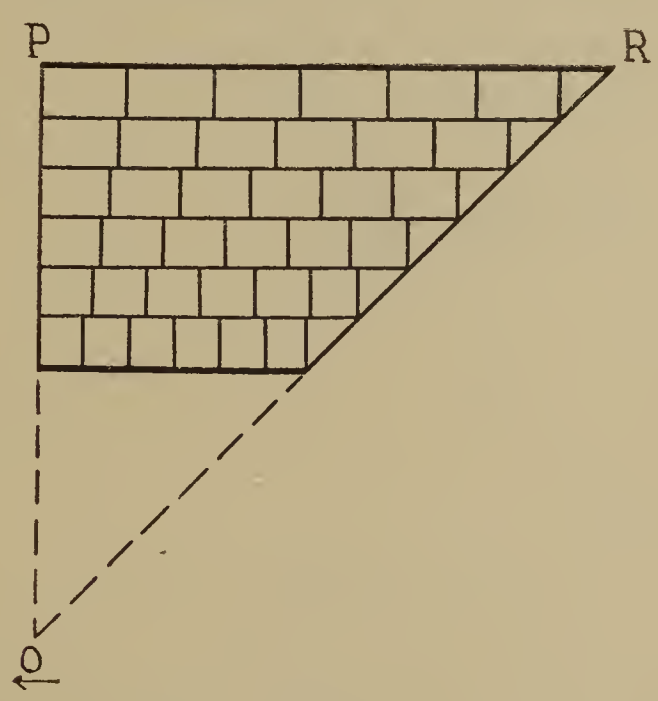

Fig. 5.

density near the edges was assumed to be the same as that over the rest of the surface.

An assumption which gives a series of flow lines approximating closely to the actual ones as represented in Fig. 4, and which makes use of perfectly insulating surfaces instead of conducting ones, is as follows:

Second Method. Determination of a lower limit for the shape factor. Let us place a perfectly insulating plane PO (Fig. 6) perpendicular to PR and parallel to the axis of the prism. Let x be the distance from the edge S to the intersection of the plane PO with the inner prism QS. Then to the left of PO the isothermal planes will be parallel, and for this part of the insulation

the shape factor will be A/t. To the right of PO we will imagine an infinite number of insulating planes passing thru O and constraining the heat to flow radially outward from O. Let us consider a plane TO making an angle θ with PO. A third plane, $T'O$, makes an angle $d\theta$ with TO. Then for the shape factor of the element included between TO and $T'O$ we have from (10), since $TO : UO :: x + t : x$,

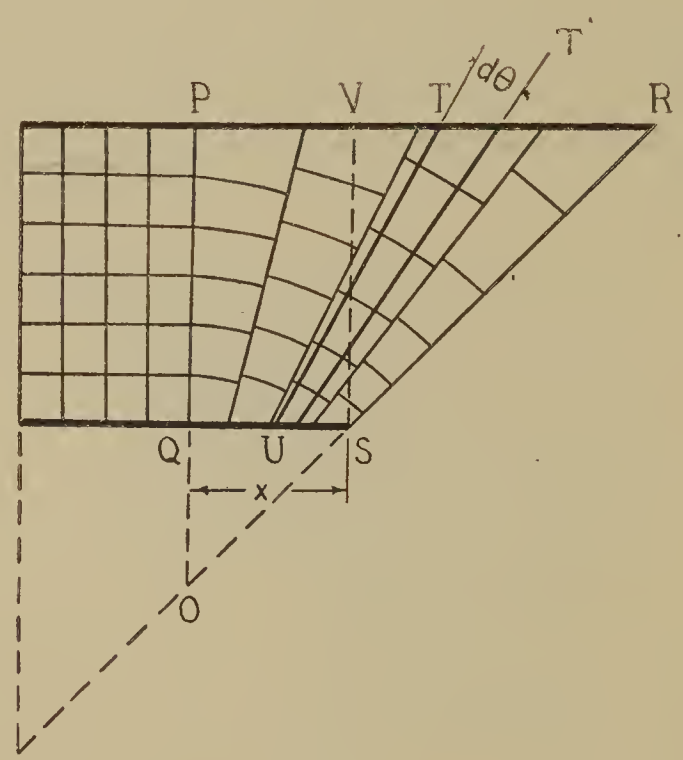

FIG. 6.

$$dS = \frac{1}{\int_x^{x+t} \frac{dr}{lr\,d\theta}} = \frac{l\,d\theta}{\log_e \frac{x+t}{x}} \tag{14}$$

whence for the whole sector POR,

$$S = \frac{\frac{1}{4}\,\pi\,l}{\log_e \frac{x+t}{x}} \tag{15}$$

If we take $x = \frac{1}{2}\,a$, and $x + t = \frac{1}{2}\,b$, then we have for the whole prism:

$$S = \frac{2\,\pi\,l}{\log_e \frac{b}{a}} \tag{16}$$

As we have seen, the shape factor obtained by this method must be too small. As a matter of fact, we find by comparing equations (13) and (16) that they differ only in the value of the coefficients. Equation (13), which is supposed to give the upper limit, has a coefficient 8, while (16), which gives the lower limit, has for a coefficient 2π or 6.283. The former is about 27 per cent. greater than the latter. Fortunately, however, we can go a step further and narrow down these limits.

Equation (15) applies to the shape factor of the sector POR, Fig. 6. For the portion to the left of PO we obtain the shape factor by the equation $S = A/t$.

On the other hand, we may apply this equation to the whole surface and then add a correction term for the effect of the edge. The shape factor S for the sector POR exceeds the shape factor for $PVSQ$ by the amount:

$$S' = \frac{\frac{1}{4}\pi l}{\log_e \frac{x+t}{x}} - \frac{xl}{t} \qquad (17)$$

Let us place $t/x = p$,

then

$$\frac{S'}{l} = \frac{\frac{1}{4}\pi}{\log_e (1+p)} - \frac{1}{p} \qquad (18)$$

From the assumption made in deriving (15), we know that the value of S' from (18) must always be too small, no matter what value we may choose for x (and hence for p). The value of S' closest to the true value will be that which is the greatest. We can obtain such a value by finding the maximum value of S' as p diminishes from ∞ to $2t/a$. To obtain this, we differentiate (18) with respect to p and place the result equal to zero.

$$\frac{d\left(\frac{S'}{l}\right)}{dp} = \frac{-\frac{1}{4}\pi}{(1+p)\left[\log_e (1+p)\right]^2} + \frac{1}{p^2} = 0 \qquad (19)$$

This reduces to:

$$\log_e (1+p) = \frac{\frac{1}{2}\sqrt{\pi}\, p}{\sqrt{1+p}} \qquad (20)$$

By solving this equation by a series of approximations, we obtain:

$$p = 4.61 \text{ or } x = 0.217\ t \tag{21}$$

Substituting this in (18):

$$S' = 0.2385\ l$$

This gives the amount by which the shape factor exceeds the value it would have if there were a perfectly insulating plane at VS. For the effect of the whole square edge, we must double this, and so obtain

$$S_c = 0.477\ l \tag{22}$$

We can now calculate the shape factor for the whole prism by the very simple formula:

$$S = \frac{A}{t} + 0.477\ \Sigma\ l \tag{23}$$

Here A is the total area of the *inner* prism, t is the thickness of the insulation, and $\Sigma\ l$ is the sum of the lengths of the edges of the prism. It must be remembered that in calculating the maximum value of S' in (17), we can vary x only from 0 up to $\frac{1}{2}\ a$. If, therefore, $0.217\ t$ (see 21) is greater than $\frac{1}{2}\ a$, that is, if the thickness of the insulation is more than 2.3 times as great as the side of the inner prism, then equations (22) and (23) do not apply, but equation (16) should be used. This case, however, rarely occurs in practice, for it implies a furnace with outside dimensions more than 5.6 times the inside dimensions.

From its method of derivation we know that, except with furnaces of exceptionally thick insulation, equation (23) will be much more accurate than the more complicated equation (16). The true value of the shape factor must, however, be greater than that given by (23), but less than that given by (13).

The method of deriving equation (21) has shown us that the effect of an edge in disturbing the flow lines extends back only a relatively short distance from the edge. Thus with a layer of insulation one inch thick, the flow lines (see Fig. 4) only 0.22 inch back from the edge of the inner prism are already practically perpendicular to the surfaces of the prisms. For this reason an equation of the form of (27) is certainly much more accurate than one of the form of (13) in all cases where the thickness

of the insulation is not more than twice the distance between two edges of the inner prism. This equation (23), besides having the advantages of accuracy and simplicity, is more generally applicable, since it applies to rectangular prisms of all shapes (with the above limitations of thickness of insulation).

As above mentioned, the coefficient 0.477 must be too low. The true value, however, can be determined by experiment. In the experimental part of this paper the results of such a determination are given.

(5th Case) Cubes: Let us apply the two methods we have used for the prism to the case of the cube. In this way we will obtain an upper and a lower limit for the shape factor.

We will consider the case of a cube (with side a) covered with a uniform thickness, t, of insulation, so that the outside surface of the insulation is also a cube (of side b). Let A and B be the total surfaces of the inner and outer cubes, thus:

$$A = 6\,a^2 \text{ and } B = 6\,b^2$$

First Method. Upper Limit for S: We assume that the isothermal surfaces are parallel to the faces of the cubes, and by a method similar to that used for the prism and sphere we obtain:

$$S = \frac{12}{\frac{1}{a} - \frac{1}{b}} = \frac{12\,ab}{b - a} = \frac{6\,ab}{t} = \frac{\sqrt{AB}}{t} \qquad (24)$$

The close similarity of these equations with those for sphere (equation 6) will be seen at once.

Second Method. Lower Limit for S: We assume that the heat is constrained to flow radially outward from the inner to the outer cube. The calculation in this case is much more complex than for the case of the prism, and will therefore not be gone into here. The result is:

$$S = \frac{7.68}{\frac{1}{a} - \frac{1}{b}} = \frac{7.68\,ab}{b - a} = \frac{3.84\,ab}{t} = \frac{0.64\,\sqrt{AB}}{t} \qquad (25)$$

The assumption that the flow takes place radially from the center of the cube is rather far from the truth, and it is therefore probable that this result is considerably too low.

Third Method: In the case of the prism the second method gave us a result (equation 16) which was identical with that found for cylinders (equation 5) of equal areas.

We might therefore obtain a lower limit for the shape factor of coaxial prisms by applying equation (5) to two coaxial cylinders of the same area as the prisms. In a similar way, for the shape factor of cubes, we may replace the cubes by spheres having the same areas, and then apply equation (6). This leads to the result:

$$S = \frac{2\sqrt{6\pi}}{\frac{1}{a} - \frac{1}{b}} = \sqrt{6\pi}\ \frac{ab}{t} = \sqrt{\frac{\pi}{6}}\ \frac{\sqrt{AB}}{t} \qquad (26)$$

$$= 0.724\ \frac{\sqrt{AB}}{t}$$

This result is higher than that (25) obtained on the assumption of radial flow. We believe, however, that this result, like that obtained for the prism by this method, is to be considered as a lower limit, altho we have not been able to find rigorous mathematical proof that such is the case.

The experiments, however, described in the experimental part of this paper have shown that the true shape factor for even very small cubes in large ones is greater than that given by equation (26).

In the rest of this paper we shall therefore look upon the results obtained by substituting spheres or cylinders of equal area as being a lower limit for the shape factor.

In the case that the thickness, t, of the insulation is not very large compared to the dimensions of the inner cube, we know that the effect of the edges and corners will not extend very far, so that over the larger part of the faces the isothermal lines will be parallel. Just as in the case of the prisms we may divide the surface up into parts. We thus calculated the shape factor by the formula:

$$S = \frac{A}{t}$$

and then apply as in the case of the prisms a correction for the effect of the edges of the form:

$$0.477\ \Sigma\ l$$

Finally we need to apply another correction for the corners (intersection of three edges). We can calculate the term giving the effect of the corners as follows:

Consider a small cube (of side X) at one of the corners of the inner cube. From the inner corner of the small cube extend the planes which form the faces of the small cube outward until they intersect the faces of the larger outer cube. This forms a new cube with side $X + t$ in the corner of the large cube. We will now calculate the shape factor between the small cube (side X) and the cube with side $X + t$.

Since only three of the faces of these cubes transmit heat, we have by (26):

$$S = 0.724 \frac{\sqrt{AB}}{t} = 2.172 \frac{X(X + t)}{t} \qquad (27)$$

The shape factor for the faces of the small cube is by (4):

$$S = \frac{3 X^2}{t}$$

And for the edges:

$$S = 0.477 \times 3 X = 1.431 X$$

The correction for the corner is therefore:

$$S_c = 2.172 \frac{X(X + t)}{t} - \frac{3 X^2}{t} - 1.431 X \qquad (28)$$

$$= .741 X - .828 \frac{X^2}{t}$$

The maximum value of this will be closest to the true value, and the condition for the maximum is:

$$\frac{dS_c}{dX} = 0.741 - 1.656 \frac{X}{t} = 0 \qquad (29)$$

Whence:

$$\frac{X}{t} = 0.448 \qquad (30)$$

Substituting this in (28):

$$S_c = 0.166\, t \qquad (31)$$

From equation (30) we see that the maximum value for S is obtained if we assume that the divergence of the heat paths

caused by the presence of a corner does not extend to more than 0.45 t back from the corner of the inner cube. In all cases where the distance between two adjacent corners is more than 0.9 t, we can apply (31) for the effect of the corner.

Similarly, we may apply this equation equally well for any rectangular shaped bodies covered with a uniform thickness of insulation, provided the distance between two adjacent corners is not less than 0.9 of the thickness of the insulation.

The following formula gives a lower limit for the shape factor of any rectangular shaped body covered with a uniform thickness, t, of insulation:

$$S = \frac{A}{t} + 0.477 \ \Sigma \ l + 0.166 \ nt \qquad (32)$$

Here A is the total area of the body. $\Sigma \, l$ is the sum of the lengths of all its edges, and n is the number of corners.

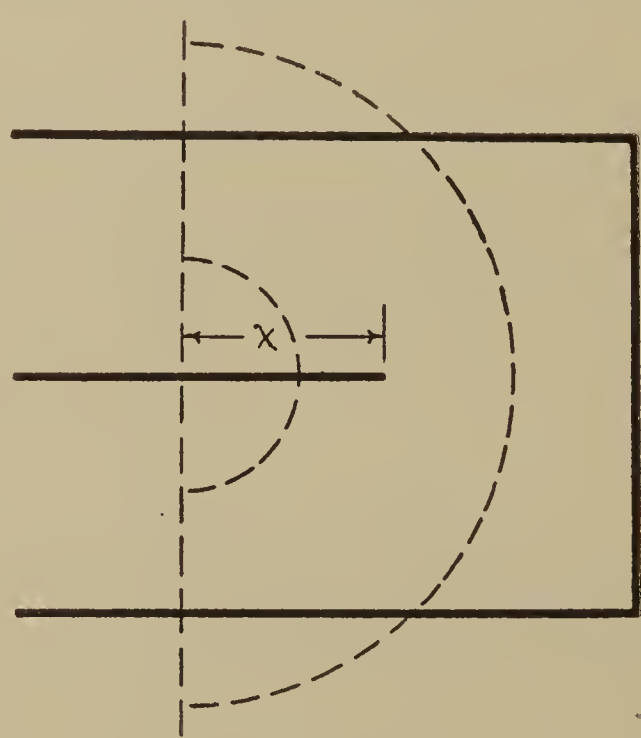

Fig. 7.

6th Case. Rectangular Plates: We have seen in the case of the prism that we must apply different formulas according as the thickness of the insulation is more than or less than 2.3 times the side of the prism. This is due to the mutual effect of the edges. Similarly we have different formulas for cubes according to the thickness of the insulation.

In the case of a thin rectangular plate, the edges and corners occur in pairs, the distance between adjacent edges being much

smaller than the thickness of the insulation, but the distance across the plate may be much greater than the thickness of the insulation. We have then to consider the "plane edge" and the "plane corner."

Plane Edge. We will define the plane edge as the straight line bounding a plane surface. Adopting a method similar to that used in the case of the cube, we replace a strip of the plane on each side of the edge by a half cylinder of the same area. Similarly, we replace the corresponding part of the outer surface of the insulation (Fig. 7) by an equivalent half cylinder. We thus obtain from (5) by similar methods to those used previously, the following expression for the effect of the plane edge:

$$S = \frac{\pi}{\log_e \left(1 + \frac{2t}{X}\right)} - \frac{2X}{t} \tag{33}$$

The condition for the maximum is:

$$\frac{dS}{d\left(\frac{t}{X}\right)} = 0$$

Carrying out the indicated operating and solving the resulting equation, we obtain:

$$X = 0.44\ t$$

Substituting this in (33), we obtain:

$$S_e = 0.952\ l$$

The coefficient here is almost exactly double that for the plane edge (see 22), so that the effect of the plane edge is practically the same as that of two edges.

Plane Corner. In an entirely similar manner, but by the substitution of equivalent quadrants of spheres instead of substituting half cylinders, we find for this:

$$X = 0.49\ t \tag{34}$$

and for the effect of the plane corner:

$$S = 0.151\ t \tag{35}$$

II. Experimental Determination of the Shape Factor.

The theoretical coefficients obtained for the effects of edges, corners, etc., are usually lower limits. To determine the true value of the coefficients, recourse was had to experiment. It was also desired to develop a method by which the shape factor for bodies of any shape whatever could be determined by experiments on small models.

There are several methods which might be used to determine the shape factor, but the most convenient seemed to be the measurement of the electrical resistance of an electrolyte placed between bodies of the desired shape.

For this purpose various shapes were constructed of glass and copper plates fastened together at their edges by Khotinsky cement. The electrolyte finally adopted was a saturated solution of copper sulfate containing 0.5 percent by volume of sulfuric acid.

A standard decimeter cube with two opposite sides of copper was used as a standard resistance.

The shape to be tested and the standard cube were filled with the same electrolyte and placed in series, so that the same constant current of 0.5 ampere flowed thru them both. By a specially devised commutating switch with mercury contacts, the current thru both shapes could be reversed at will and they could alternately be connected to a voltmeter which read the potential between the plates. Since the shape factor for the decimeter cube was known, that for the shape under test was found by direct proportion.

The shapes chosen for test represented elementary parts of cubes, prisms, cylinders, planes, etc. In each case, by varying the depth of the electrolyte in the cell, the effect of the proximity of edges and corners could be tested.

Care was taken to avoid errors due to polarization of the cell, and this proved to be easy of accomplishment. Another effect, however, described by J. S. Sand and Thom. Black (Zeit. Phys. Chem. **70**, 496 (1910)) as "Ubergangswiederstand," was very much more troublesome, and limited the accuracy of the results to about 1 to 3 percent. This effect is an apparent polarization, which is independent of the current density and which, unlike

ordinary polarization, reverses instantly on reversing the current. It amounted usually to about 0.05 volt.

Another difficulty was that caused by the meniscus at the surface of the electrolyte. The shapes employed were, however, large enough (usually 10 to 25 cm.) so that errors due to this source were not serious.

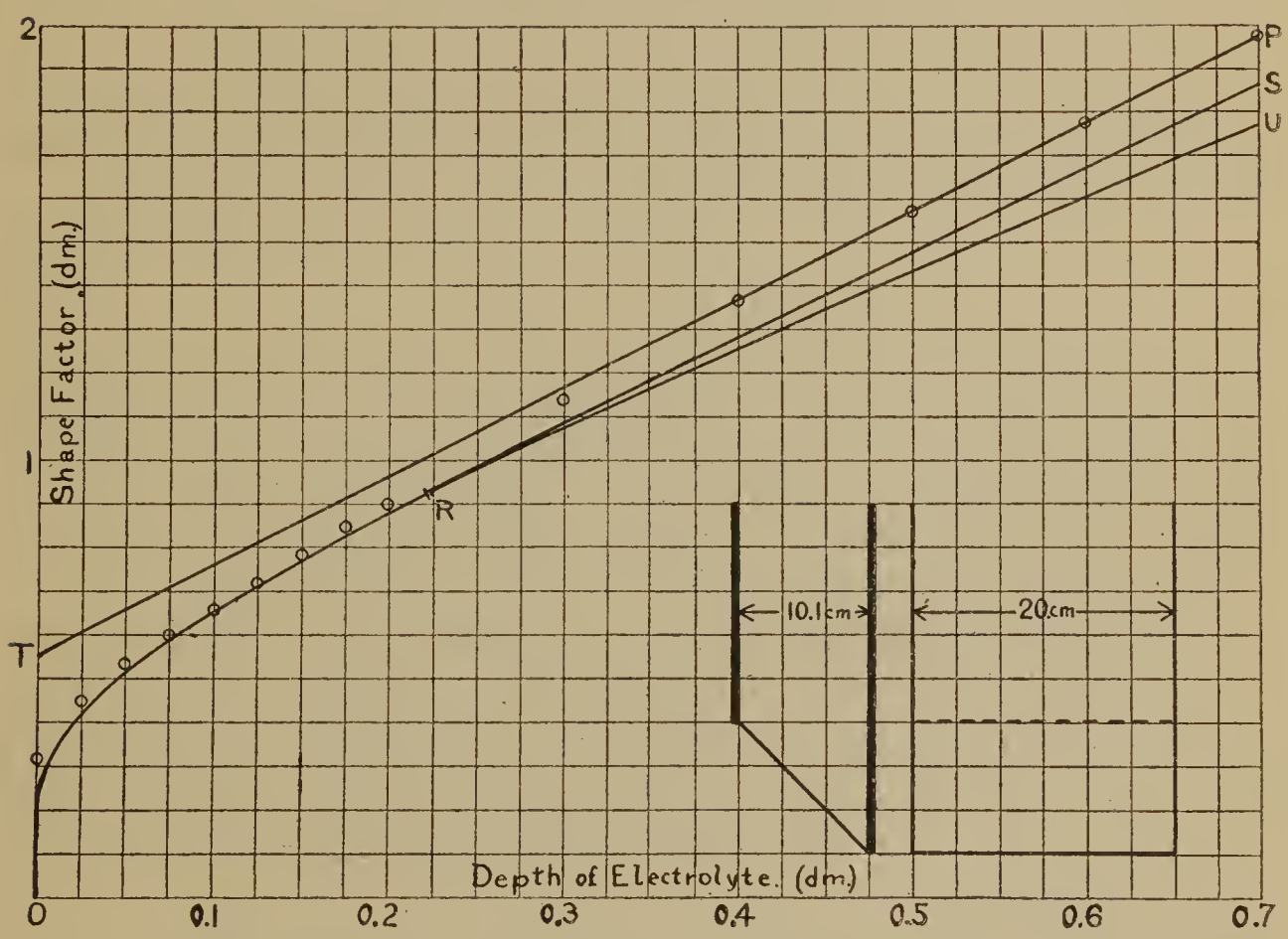

FIG 8. Shape Factor of a Square Edge.

An idea of the kind of shapes used and the data obtained may be had from Fig. 8. The small sketch gives the dimensions of the vessel used. The points give the experimentally determined shape factor in decimeters for various depths of electrolyte measured from the lower edge of the "inner" surface. The straight line PT is drawn as an asymptote to the experimental curve. The point of intersection of this line with the Y axis gives us the shape factor for half a square edge. Since the length of the edge is two decimeters we thus find for the shape factor of the whole edge:

$$S = 0.54\, l \qquad (36)$$

Our theoretical calculations had previously given us for the lower limit:

$$S = 0.477\, l \tag{22}$$

Correcting equation (23) by using the coefficient of (36), we have for the shape factor of a prism:

$$S = \frac{A}{t} + 0.54\ \Sigma\, l \tag{37}$$

Applying this to the shape used in the experiment, we have $t = 1.01\ dm$, $\Sigma\, l = 2.0\ dm$, $A = 2\ X$, whence, remembering we have only half a square edge:

$$S = 1.98\ X + 0.54 \tag{38}$$

If we compare this with the experimentally observed points, we find that it accurately represents the equation of the asymptote PT. We see that the difference between the straight line PT and the experimental points is very slight for values of X greater than $0.3\ t$. Even at $X = 0.217\ t$ (the point represented by R) (see 21), the shape factor by (37) is only 5 percent too high. On the other hand, the value given by (22) for this value of X is 5 percent too low. According to our previous calculations for values of X less than $0.217\ t$, we should employ equation (16) in place of (23). That is, we should find for the shape factor in this experiment:

$$S = \frac{\frac{1}{4}\ \pi\ l}{\log_e \frac{X + t}{X}} = \frac{1.57}{\log_e \frac{X + 10}{X}} \text{ decimeters.} \tag{39}$$

The curve URO (Fig. 8) is calculated from this equation. From our method of calculation we must consider this curve to give a lower limit. We see, in fact, that it gives values slightly too low, but below $X = 0.217\ t$ the agreement is excellent. The slight divergence at very low values of X is caused by the effect of the meniscus.

The line RS is drawn from the equation (22), and it will be seen that this line, together with RO, actually forms a lower limit for the shape factor.

In a similar way the shape factors for various other cases

were determined. The following shapes were studied experimentally:

Square edge
Square corner
Plane edge
Plane corner
Small square prism

The results obtained in this way will be given in the Summary.

Summary.

1. The steady flow of heat thru solid bodies of any shape may be calculated from the formula:

$$W = S \int_{T_0}^{T} k dT \qquad (3)$$

where W = watts of heat flow

S = shape factor, a quantity of the dimension of a length which depends only on the shape and size of the body and the position of the surfaces by which the heat enters and leaves the body

k = the heat conductivity of the body in watts per cm. per degree. If k is independent of the temperature, the integral becomes simply $k\ (T - T_0)$, and we have $W = Sk\ (T - T_0)$

2. Only in three simple cases, planes, cylinders and spheres, can S be determined rigorously by calculation. In other cases certain simplifying assumptions must be made, so that the results are of limited accuracy.

3. There are three types of assumptions that are useful in calculating the shape factor:

A. Assuming some given shape for the isothermal surfaces. This is equivalent to placing in the body perfectly conducting but infinitely thin surfaces. The shape factor calculated in this way is, of course, too high, and must be considered an upper limit. Many different assumptions may be made regarding the shape of the isothermal surfaces, and since the results will always be too high, that assumption is the best which gives the lowest result.

B. Assuming some given shape for the heat flow lines. This is equivalent to compelling the heat to flow in a given way by interposing infinitely thin surfaces of perfect heat insulating material. The shape factors calculated this way will be too low, and the best assumption is that which gives the highest value.

By applying both methods *A* and *B*, we can usually show that the shape factor must lie between quite narrow limits.

C. Very good approximate results may often be obtained by imagining parts of the surfaces of inflow and outflow of heat to be replaced by spheres or cylinders of equal area.

4. Experimentally the shape factor may be very conveniently determined for bodies of any shape by constructing small models of copper and glass and determining the electrical resistance of a saturated copper sulfate solution placed in them.

5. The values of the shape factors of heat insulation placed on bodies of various shapes have been determined both theoretically and experimentally. The results are given in the following table:

Shape Factors for Various Elementary Shapes.

Parallel Planes: $S = A/t$ where A is the area of the planes and t the distance between them. This applies rigorously when the planes are infinite in extent.

Concentric Cylinders:

$$S = \frac{2\pi l}{\log_e \frac{b}{a}} = \frac{2.73\, l}{\log \frac{b}{a}} \tag{5}$$

where l is the length of the cylinders, b the diameter of the outer cylinder and a the diameter of the inner one. This formula is rigorous for infinitely long cylinders.

Concentric Spheres:

$$S = \frac{2\pi}{\frac{1}{a} - \frac{1}{b}} = \frac{\pi ab}{t} = \frac{\sqrt{AB}}{t} \tag{6}$$

where b is the diameter of the outer, a the diameter of the inner sphere, B the surface of the outer and A the surface of the inner

sphere, t the thickness of the insulation, *i. e.*, $\frac{1}{2}(b - a)$. This formula is rigorous.

Square Edge: The shape factor is calculated for the *inner* surface up to the edge by the formula $S = A/t$, where A is the surface *inside* the insulation and t the thickness of the insulation. To this is added $0.54 \Sigma l$, where Σl is the sum of the lengths of all the square edges. The result is the shape factor for the whole insulation. The above factor 0.54 was experimentally determined. The theory gave 0.477 as a lower limit.

Square Corner: To the shape factor of the plane faces (inside the insulation) and the square edges add that of the square corners, which is equal to $0.15\ n\ t$. Here n is the number of corners and t is the thickness of the insulation.

Plane Edge: Where two square edges are so close together that their distance apart is less than one-fifth of t, the thickness of the insulation, then the shape factor is found by adding to the shape factor of the faces that of the "plane edge," which is equal to $0.93\ \Sigma l$. Here Σl is the total length of the plane edges. This factor 0.93 was determined experimentally. The calculated value was 0.95.

Plane Corner: To the shape factor of the plane faces (inside the insulation) and plane edges add that of the plane corners, which is $0.087\ n\ t$. Here n is the number of such corners and t is the thickness of the insulation. The factor 0.087 was experimentally determined. Calculation gave 0.15.

Small Square Rod: Where all the dimensions except the length are less than one-fifth the thickness of the insulation, then the shape factor is obtained from the formula:

$$S = \frac{6.4\ l}{\log_c \frac{b}{a}} = \frac{2.78\ l}{\log \frac{b}{a}}. \qquad (40)$$

where b/a gives the ratio of the surfaces of the outer and inner prism. The coefficient 2.78 was experimentally determined. Calculation shows that the coefficient must lie between 2.73 and 3.47.

Small Cube: Where *all* the dimensions of a rectangular paral-

lelopiped are less than one-fifth of the thickness of the insulation, the shape factor should be calculated from:

$$S = 0.79 \frac{\sqrt{AB}}{t} \tag{41}$$

where A and B are the surfaces of the inner and outer boundaries of the insulation and t is the thickness of the insulation. The coefficient 0.79 was found by experiment. Calculation gives 0.724.

Rectangular Parallelopipeds Covered with Uniform Thickness of Insulation: The following formulas apply to all those cases where the furnace is of rectangular shape with uniform wall thickness:

1st Case. All three dimensions are greater than $\frac{1}{5}$ *t.* For this case the shape factor can be calculated simply:

$$S = \frac{A}{t} + 0.54 \Sigma l + 1.2 t \tag{42}$$

2nd Case. Two dimensions greater than $\frac{1}{5}$ t and one dimension less than $\frac{1}{5}$ t. We then have:

$$S = \frac{A}{t} + 0.465 \Sigma l + 0.35 t \tag{43}$$

3rd Case. One dimension greater than $\frac{1}{5}$ t and two dimensions less than $\frac{1}{5}$ t. We have:

$$S = \frac{2.78\, l}{\log \frac{b}{a}} \tag{40}$$

as explained under the heading "Small Square Prism."

4th Case. All three dimensions less than $\frac{1}{5}$ t:

$$S = 0.79 \frac{\sqrt{AB}}{t} \tag{41}$$

as explained under the heading "Small Cube."

General Formula for the Cube. For the cube a single formula can be made to cover all four of the above cases:

$$S = \frac{6\, a^2}{t} + 6.5\, a + \frac{1.2\, t}{\sqrt{1 + 0.16 \frac{t^2}{a^2}}} \tag{44}$$

where a is the length of the side of the cube and t is the thickness of the insulation covering it.

6. Any actual problem may usually be solved by considering the insulation made up of various parts for each of which the shape factor is given in the table. An actual problem will illustrate.

Given a rectangular electrically heated oven, with inside dimensions 8 x 10 x 20 inches. The walls are of a uniform thickness of 8 inches. This makes the outside dimensions 24 x 26 x 36. The shape factor is calculated from the formula:

$$S = \frac{A}{t} + 0.54 \Sigma l + 0.15\, n\, t \qquad (45)$$

Here A is the area of the inside of the wall of the oven. This is 880 sq. in., thus:

$$\begin{aligned} & 2 \times 8 \times 10 = 160 \\ + & 2 \times 10 \times 20 = 400 \\ + & 2 \times 20 \times 8 = 320 \end{aligned}$$

The thickness of insulation is 8 inches. Hence, $A/t = 110$. The sum of the lengths of the edges $\Sigma\, l$ is 152 inches, thus:

$$\begin{aligned} & 4 \times 8 = 32 \\ + & 4 \times 10 = 40 \\ + & 4 \times 20 = 80 \\ \hline & 152 \times 0.54 = 82 \end{aligned}$$

The third term $0.15\, n\, t$ is 9.6, whence the shape factor is the sum of the three terms:

$$\begin{array}{r} 110 \\ 82 \\ 9.6 \\ \hline 201.6 \end{array}$$

It is interesting to compare this result with that obtained by the usual methods.

The most common method is to calculate the shape factor from the formula $S = A/t$, where A is the arithmetical mean of the

inner and outer surfaces of the oven. The outer surface is 4,848 square inches. This would give for the shape factor:

$$S = \frac{808 + 4848}{2 \times 8} = 353.5$$

This is very different from the 201.6 obtained by our new method, and well illustrates the serious error that may be made by using the ordinary rough method.

If the geometric mean be employed instead of the arithmetic, the result is:

$$S = \frac{\sqrt{808 \times 4848}}{8} = 247.4$$

This is much better than 353, but is still quite different from 201.6, being 23 percent too high.

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DISCUSSION.

E. F. Northrup (*Communicated*): In reading this paper one is impressed with the scholarly treatment the writers have given a complex but scientifically and commercially important subject. The flow of heat is often difficult of calculation, and *always* difficult of precise measurement. The presentation given in this paper has greatly smoothed out the first of these difficulties. It is clear, logical and understandable, and the formulæ given by the writers can, with their suppositions, be applied to concrete cases without difficulty.

The writers have contributed so much new material and have so clearly revealed their strong grasp of the subject that one is led (human nature being as it is) into regrets that they have not done more. The experimentalist, especially, wishes that they

had given a full theoretical treatment and numerical calculations of at least one concrete case of moderate complexity, of actual heat flow. One would like to see the formulæ for the flow of heat outward thru the walls of a hollow cube applied to such a cube built of fire brick and one built of metal and then have these calculations (which would serve as a guide to those less skilled in the making of calculations) compared with results experimentally obtained from actual constructions. I believe this additional effort of the writers would have added much to our understanding of the full usefulness of the formulæ and that new information of great value would have been obtained. The Society, however, owes its indebtedness to the writers for the two dozen pages of sound theory which has been developed and which henceforth must form the starting point in any practical calculations or scientific tests in the difficult subject of heat flow.

The object in determining the function designated the "shape factor" is, evidently, in the minds of the writers, to permit the making of more accurate calculations of the flow of heat thru furnace walls. In fact, the first part of the title of the paper is "Flow of Heat Thru Furnace Walls." The writers ignore, however, any application of the general equation, when including a shape factor of determined form, to a calculation of heat flow.

The entire paper is concerned only with the determination of this function for such important geometrical shapes as parallel planes, concentric cylinders, concentric spheres, rectangular parallelopipeds and the cube. The reader is left with the impression that if the values of the shape factor S, summarized on pages 20 to 22, are substituted in the general equation (2), page 2, that the heat flow from an actual furnace can be calculated to a high precision. The writers fail to point out the relative importance of the shape factor and other factors which must be known, or guessed, when the true rate of loss of heat from an actual furnace is to be calculated. It is easy to show that in very many cases there are other factors, as surface resistances, variable and uncertain conductivities, departures from the state of temperature equilibrium, heat losses by the outflow of hot gases, etc., which have such large relative importance that it is immaterial that the shape factor be known to a precision as close to the true value as 23 percent. (Example, pages 75 and 76.)

If equation (2), page 54, is solved for the conductivity k it is observed that, as the quantities W and T are quantities which it is possible to measure with accuracy, the value of k becomes known if S is known. The determinations which the authors have made of the values which the shape factor takes for various geometrical forms cannot fail, therefore, to be of value in the important problem of experimentally determining thermal conductivities of heat insulators.

But here, again, other factors are involved which have large relative importance, and the writer has been induced by a study of this excellent paper under discussion to offer a brief separate contribution at this meeting which treats the question of measuring conductivities and the factors which must be considered to obtain results of value.

The following formulæ are sometimes useful when it is required to determine the temperature of any point in the insulation, the temperature of two other points being known.

First, case of *Parallel Planes:*

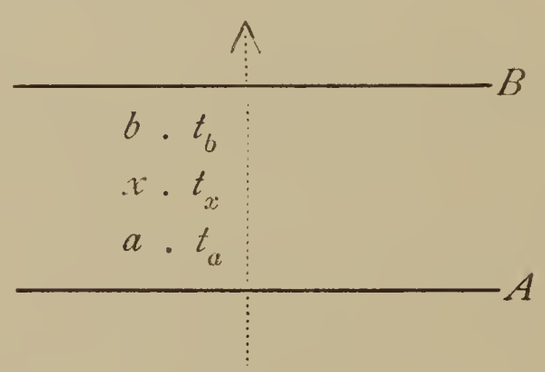

Fig. 1.

Assume that heat is flowing in the direction of the arrow (Fig. 1) between the parallel planes A and B, and that the temperatures t_a at a point distant a from plane A and t_b distant b from plane A are known. Then the temperature t_x at a point x distant x from plane A is:

$$t_x = \frac{x\,(t_b - t_a) + t_a b - t_b a}{b - a} \qquad (1)$$

If a is taken at the plane A, equation (1) reduces to:

$$t_x = \frac{x}{b}\,(t_b - t_a) + t_a.$$

Second, case of *Concentric Spheres:*

Assume that heat flows radially from the surface of the inner spherical surface A to the outer spherical surface B. (Fig. 2.)

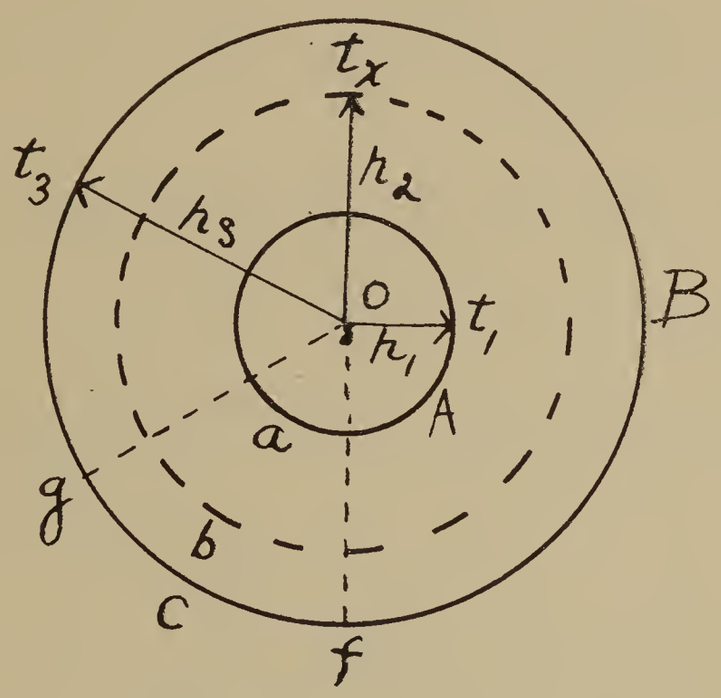

FIG. 2.

Assume that the insulating material has a constant conductivity k or resistivity $\rho = 1/k$.

Assume that at the time the temperatures are taken, equilibrium temperatures have been attained.

The problem is to determine the temperature t_x at radial distance r_2 from center O, when the temperature t_1 at radial distance r_1 and the temperature t_3 at radial distance r_3 are known.

The flow of heat along any cone as $O\ f\ g$ is:

$$W \propto \frac{t_1 - t_x}{R_1} \tag{2}$$

where R_1 is the thermal resistance along the cone from surface a to surface b. Similarly for the same cone between the surfaces b and c,

$$W \propto \frac{t_x - t_3}{R_2} \tag{3}$$

where R_2 is the thermal resistance from surface b to surface c.

Now,

$$R_1 = \frac{r_2 - r_1}{r_1 \, r_2} \, k \text{ and } R_2 = \frac{r_3 - r_2}{r_2 \, r_3} \, k,$$

where k is a constant.

Equating equations (2) and (3) and substituting the values of R_1 and R_2 we obtain:

$$t_x = \frac{t_1 \, r_1 \, (r_3 - r_2) + t_3 \, r_3 \, (r_2 - r_1)}{r_2 \, (r_3 - r_1)} \tag{4}$$

Third, case of *Concentric Cylinders:*

The formula to be given is rigid if the cylinders are of infinite length and the thermal conductivity is strictly constant.

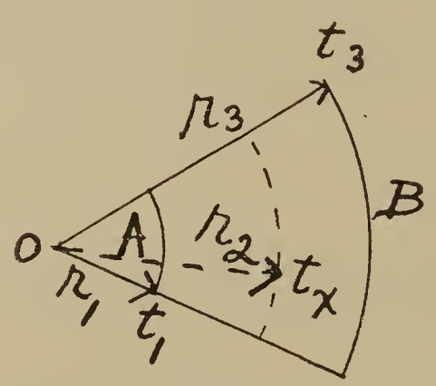

FIG. 3.

In Fig. 3 is shown a sector of a hollow cylinder having the inner surface A and the outer surface B.

The temperature t_1 at radial distance r_1 and the temperature t_3 at radial distance r_3 from the axis O are given, and it is required to find the temperature t_x at the radial distance r_2. Then, by reasoning similar to that employed in the second case, we have:

$$t_x = \frac{t_1 \log_{10} \frac{r_3}{r_2} + t_3 \log_{10} \frac{r_2}{r_1}}{\log_{10} \frac{r_3}{r_2} + \log_{10} \frac{r_2}{r_1}} \tag{5}$$

Equations (4) and (5) were deduced to solve practical cases which arose in connection with the writer's research work.

As an example of the use of equation (5), take the following case, which applies to a tubular furnace constructed and used by the writer:

$r_1 = 1.5$ inches $\quad t_1 = 1420$ °C. $\quad \log_{10} \frac{3}{1.5} = 0.301$

$r_2 = 3$ " $\quad t_3 = 200$ °C.

$r_3 = 7$ "

$\log_{10} \frac{7}{3} = 0.3674$

Then the temperature at any point 3 inches from the axis is:

$$t_x = \frac{1420 \times 0.3674 + 200 \times 0.301}{0.3674 + 0.301} = 870.57 \text{ °C.}$$

Palmer Physical Laboratory,
Princeton University,
Princeton, N. J.,
July 15, 1913.

J. W. RICHARDS: I wish to make a few remarks about the method which Dr. Langmuir has used to measure thermal resistance by means of electric resistance. The fundamental assumption of condition of these electric measurements is, as it strikes me, first, that the two plates are practically at the same potential all over, that is, that they have equi-potential surfaces. But in the case of radiation from a furnace with a shape that is anything but a sphere you do not have an equi-thermal surface on the outside, so the analogy does not strike me as exact enough for the inferences which are drawn from it by the authors. When heat is passing from the inside of a cube to the outside, we know by experience that the center of the faces of the cube are hotter than the center of the edges, and the center of the edges are hotter than the corners. So you have a variation of temperature over the surface and any side of the cube is hottest in the middle and coolest at the corners. Now, that condition you do not have when you make the electric experiment of taking the two plates and passing a current across and measuring the resistance between them, because each of the two plates has practically—I won't say theoretically, but practically—the same potential at all parts of the surface. Therefore, you have a condition which corresponds, I think, to the first assumption of Dr. Langmuir that the

surfaces of the cube are equi-potential surfaces. His first assumption is on the theory that they are equi-potential; and, when you make the electrical measurements, you are measuring the difference between equi-potential surfaces, so that his measurements should agree more fully with the first assumption. He gives the inference that his measurements come in between these two extremes, but I think they should duplicate the first assumption that the surfaces measured are equi-potential surfaces. Therefore, when he says at the close of his paper that while the arithmetic mean for the surfaces of the cube is 353 and the geometric mean 247, and that the geometric mean is 23 percent higher than 201, calculated from his formula, I think the statement that the geometric mean 247 is 23 percent too high is putting too much value on his experimental data, and it may be that his experimental data are 23 percent too low. I should like very much to see Dr. Langmuir take Prof. Northrup's advice and construct some of these shapes out of one given material and actually measure the heat flow in order to check his results. I do not think there is much reliance to be placed on them until they have such a trial.

F. A. Lidbury: I do not quite understand the point of your criticism. Is it that an isothermal surface is practically unobtainable?

J. W. Richards: No, but the conditions of using the equi-potential surface do not exist, except for the surface of a sphere. Taking particularly the example of a cube which is radiating heat from the outside, you certainly do not have an equi-potential surface.

F. A. Lidbury: Why is it necessary to introduce the question of radiation if you have a surface bounded by a good thermal conductor through which the heat is taken?

J. W. Richards: I must confess I have in mind the radiation of heat, in practice, from the outside of furnaces.

F. A. Lidbury: It seems to me you are introducing another element that should not be considered in connection with a paper which deals with first principles.

J. W. Richards: The paper deals with first principles, it is true. The author illustrates them by the use of these different

shapes, one of which, a cube, such as I have experimented with myself. He gives this as an illustration of the application of the principles, but it is only under very limited conditions or circumstances that you would have such conditions as are assumed here, namely, a cube with the outside surfaces at one equi-potential temperature.

F. A. LIDBURY: Even considering such problems in relation to furnace practice, you have got to get down definitely to the various factors which you are dealing with, and in the first place to consider the case of isothermal surfaces. If you want to introduce any other disturbing factors at the surfaces they should be considered by themselves. If we are going to mix up that sort of thing with the measurement of thermal conductivities we shall never get anywhere.

C. W. COMSTOCK: If I understand Dr. Richards correctly, I do not think that the criticism he has made is applicable to the paper.

In the first method which Mr. Langmuir describes, and which he applies on pages 56 and 57 to rectangular prisms, he states explicitly that he has assumed the inner and outer surfaces of the rectangular prisms, as well as all planes parallel to them, to be isothermal surfaces. However, in the second method, by which he derives the value which he describes as the lower limit of the shape factor, and which begins near the bottom of page 59, he very distinctly does not assume that the outer face of the prism is an isothermal surface, and in Fig. 4 he indicates by a sketch what he considers as the approximate forms of the isothermal surfaces.

I may have misunderstood Dr. Richards, but I thought he objected that the assumption of uniform temperature over the outer surface of a cube was not justified. That assumption is not made in the second method of calculation.

J. W. RICHARDS: My point was particularly this: That when Dr. Langmuir made the electrical measurement to get by analogy the shape factor, he was duplicating his first assumption, since the surfaces electrically were equi-potential, and that his experimental result, if it was equally accurate, should have agreed with his first assumption and not have dropped in between. The

inference is made that his experimental determination would give him the real thing and drop in between these two extremes, but his experiment duplicated the conditions of his first assumption. I have the further criticism to make that he says that he was troubled by the meniscus; it seems to me he could have easily avoided that by closing the cell and getting rid of the meniscus altogether. As it is, his experimental values duplicate his first experiment, with small inaccuracies as regards the meniscus, but he does not realize the geometrical weight which he gives them, that is, assuming that his experimental values are equally correct. I think it is probable that the geometric mean is quite as likely to be true as the experimental results.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Denver, Colorado, September 9, 1913, President E. F. Roeber in the Chair.

SOME ASPECTS OF HEAT FLOW

By Edwin F. Northrup.

The analytical theory of the movement of heat was given by Fourier, in 1822, in one of the most masterly treatments of mathematical physics ever written. Accepting the assumption of a constant thermal conductivity for any particular case, which Fourier thought best to make, one can find in his treatise differential equations and methods of solving them which will serve for the solution of any heat problem to which it is practicable to apply analysis. But the advance in experimental science and the commercial requirements of recent years have developed phases of the problem of heat flow and demands for precise thermal measurements of which Fourier was completely ignorant. Any one, however, whose business it is to deal with heat problems cannot afford to be ignorant of the wealth of fundamental theory contained in Fourier's "Théorie Analytique de la Chaleur."

It is of some of the very modern aspects of heat flow and of a method of viewing heat problems, which I advocate, that I wish to speak.

There is no way of viewing a problem more illuminating than seeing it in its various phases thru the true analogies which it bears with similar problems. Especially in the study of physical science, a fruitful brain economy and a clear perception of new aspects are best established by mentally associating phenomena which belong in groups and classes according to the natural physical connections or analogies which exist among them.

A physical analogy is a very definite thing, susceptible of precise definition. I have considered the precise meaning and "The Use of Analogies in Viewing Physical Phenomena" at length in a paper printed in July, 1908, in the Journal of the Franklin Institute. I shall now treat some heat problems according to the principles there considered.

General Equations Expressing Analogies.

"From a general mathematical point of view, all phenomena may be considered as being the relations which maintain between quantities used to express causes and effects by which the phenomena are characterized."[1]

Consider the relation

$$W \frac{da}{dx} = F_1 + F_2 + F_3 + \text{etc.} \qquad (1)$$

"The mathematical form of this equation indicates that a certain magnitude a varies with respect to another magnitude x and that this variation of a with respect to x is proportional to the sum of the quantities F_1, F_2, F_3 – – etc." The quantity da/dx is here assumed to be some physical effect which results from the combined action of one or more physical causes, F_1, F_2, F_3, etc. A physical *effect* may be considered as any variation with time or space of some magnitude, designated a co-ordinated; as a length, a surface, a volume, an angle, a magnetic or electric flux, a heat flux, or an energy content of any type.

A physical *cause* or physical causes which determine an effect may be classified as forces; mechanical, electrical, magnetic, gravitational, etc., and temperature differences. They may be simple forces or generalized forces in the Lagrangian sense.

In determining the analogies with heat flow, we may consider that only a single cause is acting to produce an effect. Our equation then becomes:

$$W \frac{da}{dx} = F \qquad (2)$$

Now, the element a in equations (1) and (2) may be treated either as a *primary element* or as a *derived element*. By a primary element we are to understand an element which cannot be further decomposed in thought. It is then considered as a quantity which represents an elementary concept; as a quantity of electricity, a quantity of magnetism or a quantity of heat. It is, in this case, a single co-ordinate which varies only when the co-ordinate x varies.

[1] Use of Analogy in Viewing Physical Phenomena, by Edwin F. Northrup. Jour. Frank. Inst., July, 1908, page 10.

When we consider a to be a derived element we understand it to be a quantity which is related by one or more equations of union with another element e which is then taken as the primary element. Such equation of union will have the form

$$a = \frac{de}{dx} \tag{3}$$

If there is but one equation of union, and the primary element is called e, a will have the character of velocity, namely, a flow of a primary element; as a flow of fluid which constitutes a current of matter, or a flow of electricity which constitutes an electric current, or a flow of heat which constitutes a thermal current. a is then the variation of the primary element e, from which it is mentally derived.

If a is a derived element, equation (2) takes the form, in virtue of the equation of union (3),

$$W \frac{d^2e}{dx^2} = F. \tag{4}$$

When a is a primary element I shall call W a *coefficient of restraint,* and when a is a derived element we shall call W, in analogy with mechanics a *coefficient of inertia.*

If we assume a to be a derived element, equation (4) holds, and if, further, W and F are both constant, then equation (4) means that the rate of change of the primary element is changing uniformly in space or time; that is, the effect partakes of the nature of an acceleration. Thus, integrating equation (4),

$$W\, e = \tfrac{1}{2}\, F\, x^2 + A_1\, x + A_2 \tag{5}$$

If we give to the symbols the meaning

W = mass of a particle,
F = a constant force of gravity,
F/W = g where g is the acceleration of gravity,
e = vertical distance of descent, and
x = time,

then equation (5) becomes

$$e = \tfrac{1}{2}\, g\, x^2 + A'_1\, x + A'_2. \tag{6}$$

which is recognized as the law of bodies falling in a vacuum.

It will at once be noted that developments of equation (4) lead to a class of problems which deal with rates of variation of the primary element. Heat is a quantity which does not possess inertia, nor does it simulate it in its manifestations. Modifications of equation (4) will apply to thermal problems only in those cases where the steady state of heat flow or temperature equilibrium has not become established.

Tho further attention to equation (4) would lead on to many interesting and valuable deductions, we must hold ourselves to a consideration of equation (2). We shall then be confined to thermal analogies in which the assumption is made that the effect produced by a single steadily acting cause has itself become constant.

Heat Flow Analogies.

Returning to equation (2), assume that a is a primary element, and that its variation with the co-ordinate x is the so-called effect which is brought about by the action of a single constant cause F. This cause may be a constant mechanical force, a constant electromotive force or a constant temperature difference. Also assume, for the moment, that W is a *constant* coefficient of restraint. W may be a frictional resistance, an ohmic resistance or a thermal resistance. Then, as W and F are constant, da/dx is constant.

Call $da/dx = k$, and we have the general relation:

$$k = \frac{F}{W} \tag{7}$$

Equation (7), identical with Ohm's law, merely expresses an intuitive or instinctive perception of the mind, that an effect is manifested directly as the magnitude of the cause producing it and inversely as the restraint which is present to its occurrence. Analogous phenomena of any group expressed by an equation can always be put in evidence by means of a table. Thus with assumptions of the character of those above we can write the following table:

Primary Element a	Co-ordinate x	Cause F	Coefficient of Restraint W	Effect $\frac{da}{dx} = k = \frac{F}{W}$
Quan. of matter or fluid	Time	Mechanical force	Frictional resistance	Material current
Quantity of electricity	Time	Electromotive force	Ohmic resistance	Electric current
Quantity of heat	Time	Temperature difference	Thermal resistance	Thermal current

Again, there is a very large group of phenomena in which the quantity k is itself a primary concept, so that in equation (7) k is an effect which is related in a proportional way to a cause F, and W is a coefficient which may or may not be a function of F. In all those cases where W is constant, these phenomena are strictly analogous among themselves and are loosely analogous with the group of phenomena governed by equation (2). Of the phenomena belonging to this class may be mentioned: a flux of mechanical strain, of sonorous vibration, of electric induction, magnetic induction and a flux of light. The causes F which determine the magnitudes of these effects are respectively: a mechanical stress, energy of vibration of sounding body, difference of electric potential, magnetomotive force and light producing activity of source. The restraints W to the manifestations of the effect are, respectively: x, rigidity of the material, sound opacity or absorption coefficient of the medium, the reciprocal of the specific inductive capacity of the medium, the reciprocal of the permeability of the medium and the coefficient of light absorption of the medium.

Again, in the group of effects governed by an equation of the form $k = F/W$ belong such relations as the quantity of electricity which is stored in a condenser and the quantity of magnetism which is stored in a mass of iron; a potential difference and a magnetomotive force being respectively the causes of the accumulation, and the reciprocal of specific inductive capacity and the reciprocal of magnetic permeability being the restraints to indefinitely great accumulation.

Analogies and Dissimilarities.

The perception of analogous relations between phenomena is made possible by mentally abstracting from the phenomena those elements which can be related, in the manner shown above, by equations of the same form. By following a mental process of this character the mind is naturally led to perceive, in addition to elements which are analogous, those features in the phenomena which are not analogous, and this gives new aspects of the phenomena which yield valuable information.

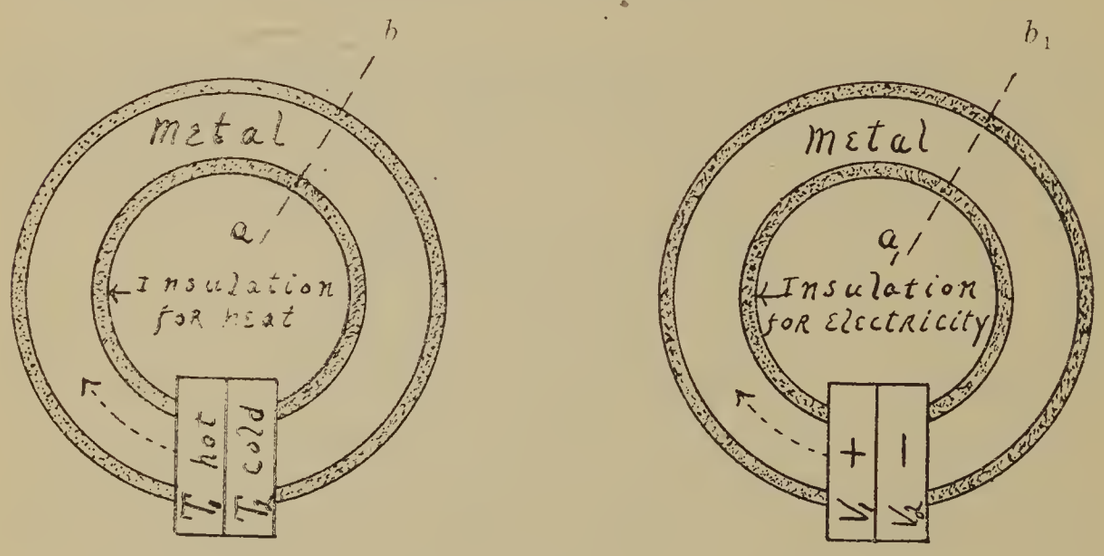

FIG. 1.

A great advantage to the progress of science in tracing the analogies between related phenomena comes from the perception to which one is led of the essential *differences* in those elements which give uniqueness to each phenomenon or group of phenomena. When these differences are perceived, their causes may be investigated and new elements and causes become discovered.

In the science of precise measurement we are led through a perception of the analogy between two groups of phenomena to adopt those methods of measurement developed for the one group and found useful. for measuring quantities which belong to the other group. In our clear perception of the unlike features in the two classes of phenomena we learn the modifications in the methods which are required.

We shall consider some of the electrical methods of measurement which are useful alike in maesuring heat flow and thermal conductivities. and we shall note where analogy fails

between flow of electricity and flow of heat. We shall then see how the methods so well adapted to measuring the flow of electricity and electrical conductivity must be modified for the precise determination of the corresponding thermal quantities.

Table of Physical Analogies and Dissimilarities Between Flow of Heat and Flow of Electricity.

Before proceeding, however, to these considerations it will be well to present in parallel columns physical statements of the chief analogies and differences between a flow of heat and a flow of electricity.

To precisely fasten the mind upon the analogies and the dissimilarities listed below reference should be made to diagrams *A* and *B* of Fig. 1. In both cases it is assumed that the steady state has been attained.

Heat	Electricity
1. Equal quantities of heat pass every cross-section a . . . b in unit of time provided the heat insulation is perfect.	1′. Equal quantities of electricity pass the cross-section a_1 . . . b_1 in unit of time.
2. The cause F of the flow of heat is difference of temperature.	2′. The cause of the flow of electricity is difference of potential.
3. The restraint W to the flow of heat is a thermal resistance—which only to a first approximation is independent of that which causes the flow, *i. e.*, temperature-difference.	3′. The restraint to the flow of electricity is an ohmic resistance, which in metals is independent of that which causes the flow, *i. e.*, potential difference.
4. Stream lines of heat flow follow paths of least thermal resistance.	4′. Stream lines of electric flow follow paths of least ohmic resistance.
5. The flow of heat is not supposed to raise the absolute temperature of the path.	5′. The flow of electricity always raises the absolute temperature of the path.
6. Heat flow is a flow of energy which is indestructible, but which diminishes in availability in the direction of lower temperature.	6′. Electric flow is a flow of electricity which is indestructible. It is only one factor of power.

Heat	Electricity
7. Direction of heat flow reversed when direction of temperature gradient is reversed, but the reversal is not generally instantaneous and often not rapid.	7′. The direction of electric flow is reversed, in ordinary cases, upon the instant of reversal in the potential gradient.
8. Flow of heat is uniform over cross-section of a bar of homogeneous material.	8′. Flow of electricity is uniform over the cross-section of a bar of homogeneous material.
9. Flow of heat produces no internal attractions or pressures in the conductor.	9′. Flow of electricity always produces centrally acting hydrostatic pressures in the path through which it flows.
10. Heat can flow in an open circuit as from center to surface of a sphere. There may be an absolute accumulation of heat. But if steady state and perfect insulation are assumed, the same quantity of heat crosses all sections of the path in the same time.	10′. Electricity always flows in a closed path. There can be under no circumstances an absolute charge of electricity.
11. No field of force exterior to a circuit of heat flow.	11′. A magnetic and an electric field of force always exterior to a circuit through which electricity flows.
12. No lines of force or temperature gradient in plane of cross-section perpendicular to heat flow.	12′. Lines of magnetic force exist in plane of cross-section of the circuit.
13. Temperature gradient follows axis of circuit.	13′. Potential gradient follows axis of circuit.
14 No thermomotive force recognized which acts upon heat to move it.	14′. An electromotive force is recognized as that which moves electricity in its circuit.
15. Heat flows are rarely observed or measured along the axis of a long linear conductor.	15′. Electric flow most generally observed and measured in long linear conductors.
16. Heat insulation of a circuit is generally very imperfect.	16′. Electric insulation of a circuit can be made practically perfect.
17. The total restraint W to heat flow is the sum of all the thermal resistances in the circuit.	17′. The total restraint to electric flow is the sum of all the ohmic resistances in the circuit.
18. Gaps and joints in the path of heat flow offer large restraints, but never open the circuit.	18′. Joints in the path of electric flow introduce restraints. Gaps in the circuit stop the flow entirely.

Further Consideration of Equation (2).

When considering the flow of heat, equation (2) becomes:

$$H = \frac{T_1 - T_2}{r} \tag{8}$$

and when considering the flow of electricity, it becomes:

$$I = \frac{V_1 - V_2}{R} \tag{9}$$

where $T_1 - T_2$ and $V_1 - V_2$ are, respectively, the difference of two temperatures and the difference of two potentials.

H is a flow of heat in watts, and I is a current in amperes. R is the total ohmic resistance between the points where the potentials are V_1 and V_2, and r is the total thermal resistance between the points where the temperatures are T_1 and T_2. Mr. Irving Langmuir has shown[2] that equation (8) can be written:

$$H = S\,k\,(T_1 - T_2) \tag{10}$$

where S is the "shape factor" of the path thru which the heat flows and k is the thermal conductivity of the material. Now, it is equally true in the electrical case that

$$I = S\,k_1\,(V_1 - V_2) \tag{11}$$

and when a problem is solved for the one case it is solved for the other.

In this connection it is to be noted that if we have derived a formula for the electrostatic capacity C of any geometrical arrangement where the specific inductive capacity between the surfaces is K, we can at once derive the shape factor S by multiplying the expression for the capacity by the factor $4\pi/K$. Thus

$$S = \frac{4\pi}{K}\,C.$$

For example, the ordinary expression for the electrostatic capacity of two concentric spheres is

$$C = \frac{K\,a\,b}{b - a}$$

[2] Flow of Heat Through Furnace Walls: The Shape Factor. This volume, p. 53.

where a and b are the radii of the inner and outer spherical shells, respectively. Multiplying both sides of this relation by $4\pi/K$, we obtain

$$S = \frac{4\pi a b}{b - a}.$$

If we put $a = d_1/2$ and $b = d_2/2$ where d_1 and d_2 are diameters, we obtain

$$S = \frac{2\pi d_1 d_2}{d_2 - d_1} = \frac{\pi d_1 d_2}{t} = \frac{\sqrt{AB}}{t}$$

where $t = (d_2 - d_1)/2$, the thickness of the insulation, and A is the surface of the inner and B the surface of the outer sphere.

Attention is now directed to the fact that the true physical analogy between the flow of heat and the flow of electricity is not as perfect as equations (10) and (11) would indicate. This is due chiefly to the fact that the conductivity k_1 of material thru which electricity flows is independent of the potential or difference of potential of any part of the path, while the conductivity k of material thru which heat flows varies more or less with the absolute temperature of the material.

This variation in k is small in the case of metals. For copper the conductivity in thermal mhos is 3.843 at 18° C. and 3.8 at 100° C., a decrease of a little over 1 percent in 82° C. But in the case of the so-called heat insulators the conductivity changes greatly for a wide range of temperature variation. In the case of electric flow the conductivity also generally changes very greatly with the temperature. But the point to specifically note is that in the electric case it is a difference of potential which determines the flow of electricity, while the conductivity of the material is only affected by temperature—a different thing than that which moves the electricity. The temperature can be controlled without changing the potential gradient. In the case of heat flow, on the other hand, temperature differences determine the flow of heat, and the conductivity will be different at the two ends of the conductor, due to the existence of the different tem-

peratures at the two ends which are required for the flow of heat to take place. In the electrical case the current or temperature of the bath affects the resistance of the conductor uniformly—or nearly so—thruout its length. Hence the change in resistance due to temperature is readily determined. In the heat case the thermal resistance changes in a very complicated manner, and we are here introduced to the first phase of heat-flow measurements, which render them vastly more difficult than the analogous electrical measurements.

Mr. Irving Langmuir has already considered so fully and so ably the various forms which equation (10) may be made to assume to meet the exigencies of heat-flow calculations that I shall omit further reference to this phase of the problem and pass at once to a consideration of the help we may derive in planning thermal measurements by considering the electrical cases and the methods of measurements which analogy suggests.

Why Measurements of Thermal Conductivity Are Difficult.

Differences of temperature are easily determined by electrical methods even when the higher temperature is 1200° C. or more, and Mr. Langmuir has shown how the shape factor can be determined for all cases which are important. Heat flows can therefore be readily calculated when the two factors, thermal conductivity and surface film resistance, are experimentally given. The experimental problem then resolves itself into (*a*) the determination of thermal conductivity over a wide range of temperature, and (*b*) the determination of surface film resistances.

The first difficulty we encounter in measuring thermal conductivity is the one already mentioned; namely, that in producing a flow of heat with which to make the measurement, by creating a temperature difference, we vary more or less the quantity we desire to measure.

The second difficulty is the extreme slowness with which temperature equilibrium is re-established after once being disturbed. It is not always easy to know when equilibrium is attained, and under the most favorable conditions long intervals must elapse

between successive observations if any change is made in the conditions. Heat measurements for this reason are always very tedious, and require much patience as well as skill.

The third and perhaps the greatest difficulty results from the impossibility of giving to any path of heat flow a thermal insulation which even approximates perfection. This difficulty increases as the thermal conductivity of the material being measured decreases.

There is a fourth difficulty less understood than the above but which if not surmounted will introduce very great errors into the measurements. I refer to the very large surface film resistance which is always found at the surfaces of thermal conductors. If, for example, one were to attempt to measure the thermal conductivity of copper by placing a heating coil in the interior of a hollow copper sphere immersed in ice water and then to take the watt input as representing the heat which flows and the difference between the temperature of the interior of the sphere and the temperature of the ice water as representing the temperature-difference, the result would give the conductivity tens if not hundreds of times too small. This is due to the extreme difficulty with which heat passes from the heated air in the interior into the body of the metal.

In the measurement of electrical resistance it is customary to clamp under binding posts the two ends of a conductor the length of which is thousands of times its diameter. The contact resistance at the binding posts is then a negligible quantity. In measuring thermal resistances, on the contrary, the form of the specimen is more nearly cubical and in the case of metals the contact resistance between the specimen and the electrodes which press two of its sides is a very large part and sometimes the greater part of the total thermal resistance from one electrode to the other.

In the light of the analogies between heat and electrical phenomena I shall now consider how the above difficulties may be in part overcome, by availing ourselves of methods of measurement analogous with the fundamental electrical methods for measuring electrical resistance.

Methods for Measuring Thermal Resistance Suggested by the Electrical Analogies.

The four fundamental methods of measuring electrical resistance are:

(*a*) By measuring the fall of potential and the current which flows,

(*b*) By comparing the fall of potential over a standard resistance and the specimen,

(*c*) By the balance method of the Wheatstone bridge, and

(*d*) By the balance method of the Kelvin Double Bridge.

In adapting any of the above methods to the measurement of thermal resistance we must, of course, always substitute for the voltmeter or potentiometer which measures a potential drop a device which measures a fall of temperature. The best device for this purpose is a thermocouple. For temperatures under 150° C. thermocouples of constantan or eureka *vs.* copper wire about No. 40 B. and S. and silk insulated may be used. For higher temperatures organic insulation must be avoided and for temperatures exceeding a few hundred degrees special alloys or combinations of the noble metals are required.

In place of the ammeter or its equivalent for measuring electrical current we have no single device which responds to rate of flow of heat. When therefore it is required to measure heat flow, it is necessary to determine the change per unit of time produced by the heat in some material substance. The quantity of water raised one degree or the quantity of ice melted in the unit of time are methods commonly employed to determine the rate at which the heat flows or is delivered. Of course the rate at which heat is produced by the source of heat can be very precisely measured and if the heat which flows through to the opposite side of the specimen is also measured the quantity of heat which does not pass between the temperature points can be determined. When the mean thermal resistance is determined by measuring the three quantities, T_1-T_2 the temperature difference, w the heat which flows in the unit of time and t the elapsed time, we employ the analogue of the electrical method (*a*) above. As this is the least precise of the electrical methods it is

even less precise in the thermal case. The time and the temperature-difference can be measured with high precision but the quantity of heat which flows through a selected path cannot. The difficulty is largely, if not chiefly, due to the impossibility of knowing the paths in which all the heat flows.

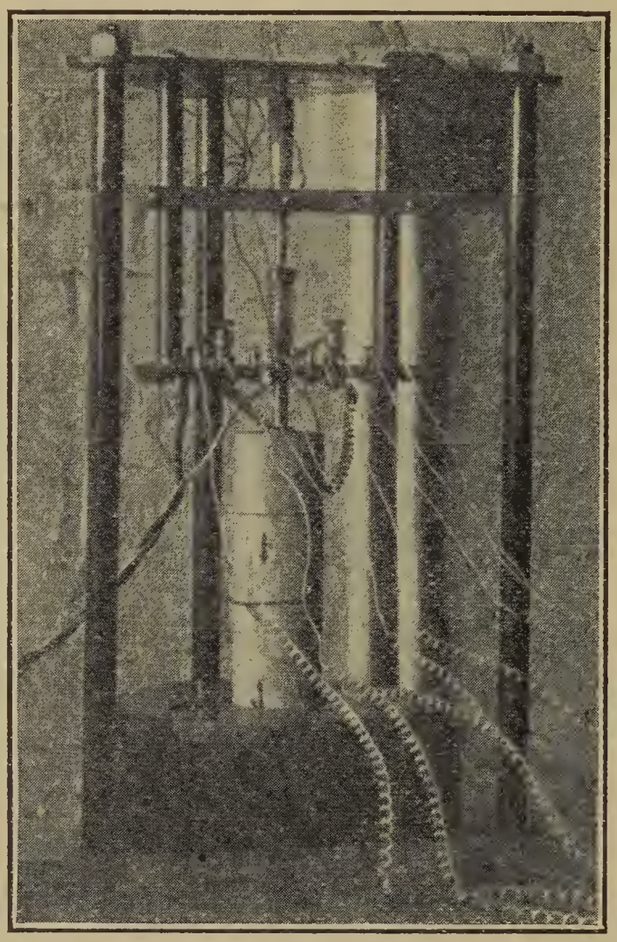

FIG. 2.

The analogue of the electrical method (*b*) suggests a method of determining a thermal resistance in certain cases which is both accurate and easy of application. As I have very carefully tested this method in its application to measuring the thermal resistance between two plane surfaces which press together, I can best bring out its advantages and show its analogy to the electrical method by giving a brief description of the actual apparatus and method employed.

Fig. 2 is a photograph of the apparatus designed by me. The principle employed in its use is based on the electrical method (*b*) and is as follows:

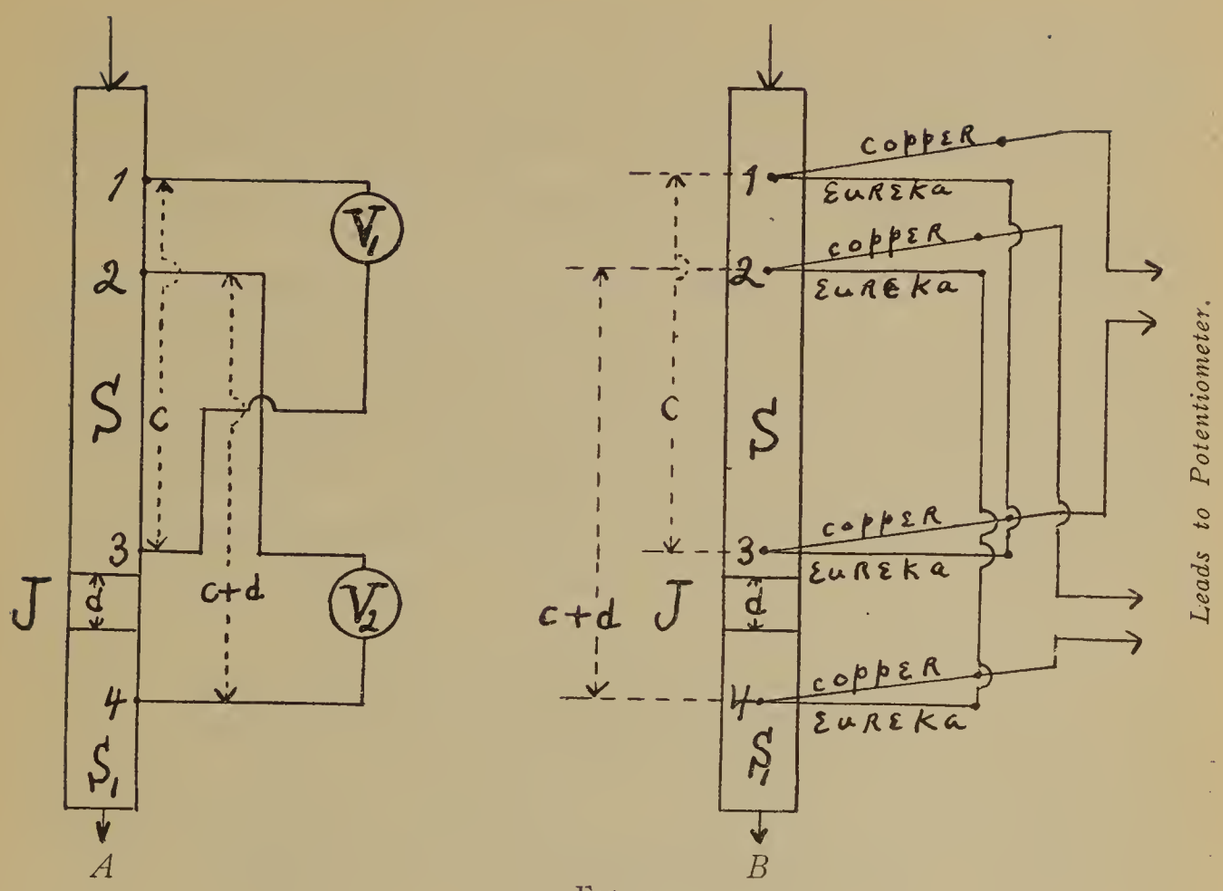

FIG. 3.

Referring to *A*, Fig. 3, let a rod of homogenous material and uniform cross-section be joined end on with another rod of like material and cross section. At the surface of union *J* let there be either a contact resistance or another short rod of different material and of length *d*.

Let 1, 2, 3, 4, be potential points so located that the length *C* of the material *S* included between points 1 and 3 is exactly equal to the length of the material *S* included between points 2 and 4. Let d_1 be the deflection of the instrument V_1 and d_2 the deflection of the instrument V_2. Both instruments must have the same constant but they need not necessarily read in volts.

Then $d_1 \propto C$ and $d_2 \propto C + x$ which gives by division

$$x = \frac{d_2 - d_1}{d_1} C. \tag{12}$$

Equation (12) gives the resistance of the length *d* of the material at *J* in terms of a length *x* of the material *S*. If *C* is taken in centimeters *x* is in centimeters. If the length *d* of the material at *J* is made zero as nearly as possible then *x* is then contact resis-

tance of the surface at J in equivalent length of the material S. This method will be recognized as the one employed by F. A. J. FitzGerald and A. T. Hinckley[3] for measuring the resistance of electrode joints and described before the Society at Atlantic City April 3, 1913.

Refer now to B, Fig. 3, and we have the exact analogy for the thermal case. The gap at J may be filled with a film of air or liquid, or the gap may be closed by pressing the piece S tightly against the piece S_1. In the former case x gives the thermal resistance in terms of equivalent length of S of a film of liquid of thickness d, and in the latter case the thermal resistance of a joint between S and S_1 for the particular pressure employed.

In the apparatus constructed and shown in Fig. 2, S and S_1 are round rods 3.8 cm. in diameter, of electrolytic copper. The ends at J are very true surfaces. The separation at J can be measured by a micrometer, and the length C is made very accurately 10 cm. The rods are carefully heat-insulated with baked lavite. The lower end is kept at zero degrees in ice water, and the upper end is heated electrically. A further description of this apparatus and the data obtained with it must be deferred to a later paper.

It may be mentioned here, however, that the thermal contact resistance of the two plane copper faces when pressed together with a pressure of 1.6 kg. is equal to 31.2 cm. of length of copper. It will be noted that the precision of the method is little affected by any small leakage of heat between the points 2 and 3, and only the difference in the leakage between the points 3 and 4 and the points 1 and 2 will much affect the measurement. As the distances 1–2 and 3–4 are very small, the former being only 0.6 cm., and as the lavite insulation is very good, errors from this heat leakage are thought to be very small.

The method of measuring thermal resistances by comparing the fall of temperature over a standard material and the material to be measured should prove a very precise one if properly carried out. The diagram, Fig. 4, suggests in outline the way I would propose to execute the method for either metallic materials or so-called heat insulators.

The standard material S should be copper for the metals and a

[3] Trans. Am. Electrochem. Soc., 23, 333 (1913).

substance like alundum of a standard grade and composition for high thermal resistance materials. G, G is a guard cylinder of the same material as the standard, and G_1, G_1 a guard cylinder of the same material as the sample X.

The results obtained should give the ratio $X/S = E_1/E_2$ with excellent precision. The proposed method is exactly analogous to the electrical method (b).

There is no exact analogy of the electrical method (c) which occurs to me as having much value in thermal measurements, but the method of the Wheatstone bridge suggested the following method for obtaining the relative thermal conductances of similar

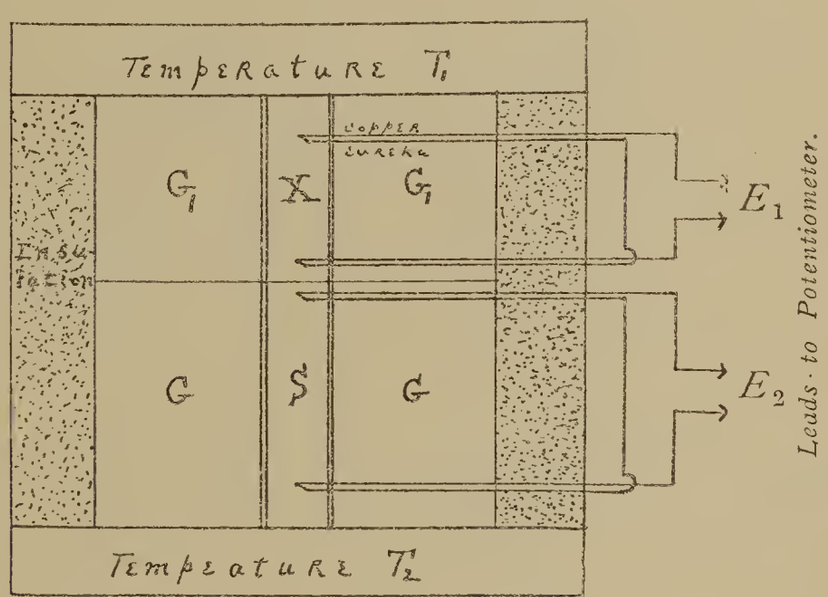

FIG. 4.

coils of wire when not impregnated and when impregnated with different insulating compounds.

The method was used as briefly described below and gave very consistent results. It in every way proved itself an excellent method for the purpose of comparing rates of heat loss from similar coils impregnated with different kinds of impregnating compounds.

Call the coils to be compared, coil A and coil B (Fig. 5). These coils must be alike in dimensions, size and kind of wire and electrical resistance at the same temperature. For minimum surface with a given weight of wire they should be wound in cylindrical form, the height of the cylinder being made equal to

the diameter of its base. The coils are connected in circuits, as shown in Fig. 5. They should be placed in still air or in a large oil bath kept well stirred with a motor. The coils are identical in form and size, and both are exposed to the same conditions for loss of heat thru their exterior surface, and it is assumed that the outside surface temperature of each will be the same. The current thru each coil is adjusted (always waiting until temperature equilibrium is established) until the resistance of each coil is the same. If the coils are wound with a large

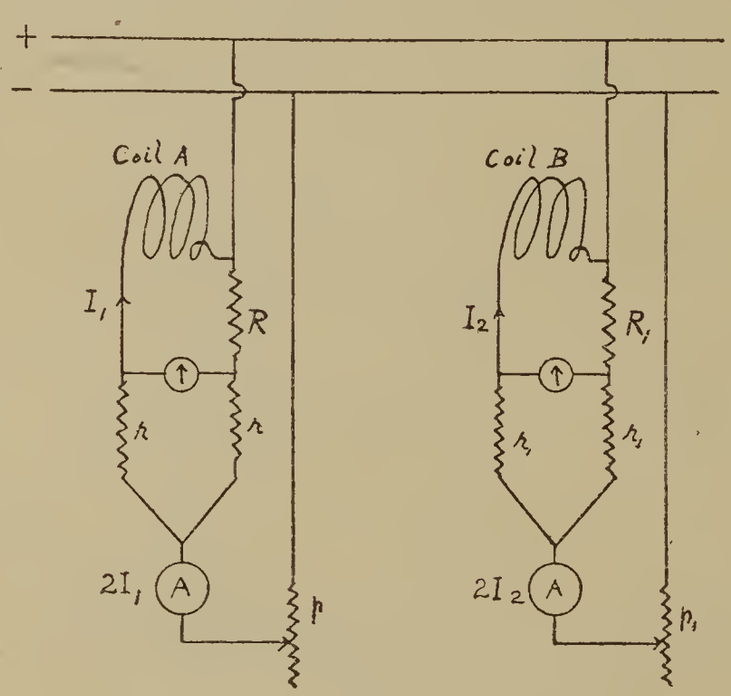

FIG. 5.

temperature coefficient wire, as copper, then when their resistances are raised by the same amount, they will have the same mean temperature. When this adjustment has been made the watts dissipated in each coil will be:

$$W_1 = I_1^2 \; R_t \text{ for coil } A \text{ and}$$
$$W_2 = I_2^2 \; R_t \text{ for coil } B,$$

where I_1 is the current thru A, and I_2 the current thru B and R_t, the resistance of each coil at mean temperature t.

The thermal conductance of the two coils will now be in the direct ratio of the watts which are supplied to each coil. Thus

$$\frac{C_A}{C_B} = \frac{W_1}{W_2} = \frac{I_1^2 \; R_t}{I_2^2 \; R_t} = \frac{I_1^2}{I_2^2}.$$

Hence it follows that for equal mean temperatures, steadily maintained for both coils, the thermal conductances will be in the ratio of the squares of the currents passed thru each coil, or the squares of the main currents supplied to each bridge arrangement.

In carrying out this method the main currents are read with a single ammeter which can be switched from one bridge circuit to the other. A single portable galvanometer which can be switched from one bridge circuit to the other serves to indicate when the bridges are balanced. The arms of the bridges which do not include a coil are made up of manganin resistances, and the main currents are adjusted by rheostats p, p_1. The resistances r, r, r_1, r_1, are all made equal, and the resistances R, R_1 are given a value which is the resistance that a coil will have when at the desired mean temperature. Four coils were intercompared by this method, with the following results: Coil No. 1 was impregnated with a compound which I shall call Y, Coil No. 2 with a compound Z, Coil No. 3 with a compound X, and Coil No. 4 was unimpregnated.

Considering No. 1 as having a conductance of 100 percent, we found:

Comparison	Percent	Average Temperature of Coil. Degrees C.	Air Temperature. Degrees C.
No. 2 / No. 1	89.1	80	23
No. 4 / No. 1	68.7	80	23.9
No. 4 / No. 1	67.5	107	23.5
No. 4 / No. 1	68.7	134	25
No. 3 / No. 1	103.2	80	23

It is of interest to note that the unimpregnated coil No. 4 had the lowest thermal conductance. The coils were cylinders wound with double cotton covered No. 20 copper wire and were 7.7 cm. in diameter and 8.3 cm. high.

I am indebted to a Princeton graduate student in electrical engineering, Mr. William Eves 3d, for testing out the above method and for very carefully obtaining the data given above.[4]

The thermal analogue of the Kelvin double bridge method (*d*) of measuring low resistances and of obtaining the conductivity of low resistance, large temperature coefficient wires in terms of a standard of conductivity (Hoopes' Bridge Method) has not been tried out by me. I have, however, tested experimentally the principle of the method and I believe it is capable of being developed into a very useful method. The diagrams *A* and *B* of Fig. 6 below are self explanatory.

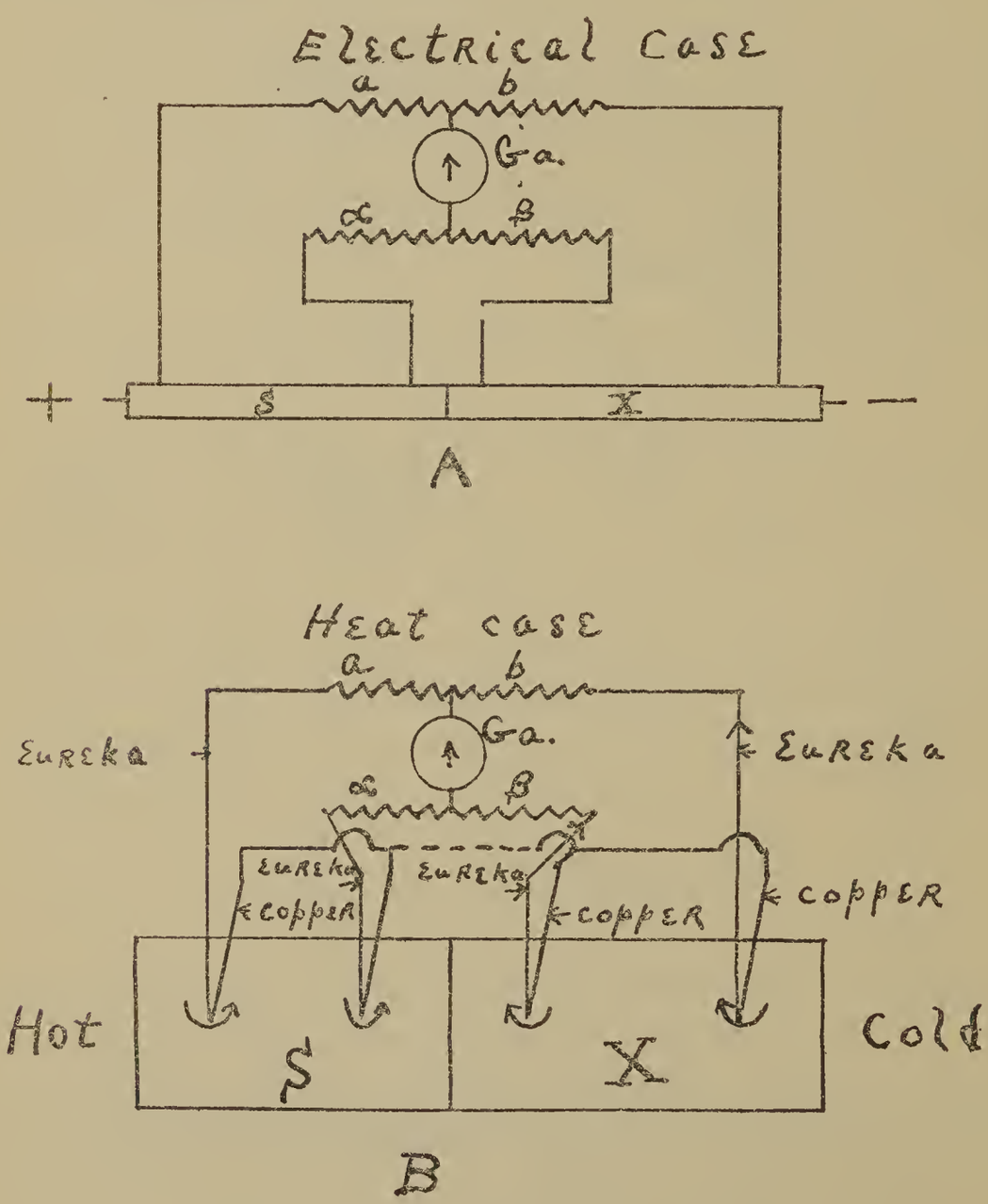

Fig. 6.

[4] See Mr. Eves' fuller description of the experiment, Metallurgical and Chemical Engineering, Sept. 1913, page 503.

In either of the diagrams A or B, the galvanometer shows no deflection and the bridge is balanced when $S/X = a/b$, provided the ratios a/b and α/β are maintained the same. The connection in B shown in dotted line may or may not be made.

In the thermal case, thermocouples are the sources of current which operate the galvanometer. The method is a zero method in every respect and it is not necessary to know the quantity of electricity or the quantity of heat which flows. By maintaining the cold end as much below as the hot end is above the surrounding temperature it is evident that heat losses from the sides of the thermal path would not introduce much error. Side guards through which the temperature gradient is made the same as that through the standard and the sample would effectually reduce this source of error.

The Use of Standards of Thermal Conductivity.

It will be at once recognized and perhaps objected to, that the above methods, taken as the analogues of the electrical methods for measuring resistance, give only *comparative* results.

But why not adopt standards of thermal resistance, and standards of thermal conductivity the same as in electrical measurements we adopt standards of electrical resistance and a standard (Matthiessen's Standard) of electrical conductivity? It seems to me that great advantages would result from this procedure. Some of these advantages are apparent from the suggestions regarding measurements given above. Others are:

The corrections for losses of heat through the insulation of the path of the heat flow, could be made negligibly small.

The principles of the Kelvin bridge, which avoids all contact resistances (surface film resistances in the thermal case) could be applied.

The measurements could all be made by accurately measuring only temperature-differences, the absolute temperature being only roughly required.

The measurements could be carried out at absolute temperatures chosen through a wide range.

The measurement of a quantity of heat is never required.

To be sure, all results would be given in terms of a ratio of the thermal resistance of the sample to the thermal resistance of the selected standard. But when by long and carefully conducted investigation the specific thermal resistance of the material selected for the standard is once obtained, all comparisons made thereafter with this standard can be expressed in the usual thermal units.

I would suggest that two standards be adopted—one for low thermal conductivity materials and one for high conductivity materials. The first might consist for example of a standard grade of alundum cement packed under a given pressure and baked to a maximum given temperature. The second might consist of 100 percent electrical conductivity copper. The thermal conductivity of this last is already pretty well known, and one careful investigation would determine the other.

I offer this suggestion in the hope it may meet with favor and prove to be as good in practice as it seems to be in theory.

Interest in the properties of matter at temperatures above those at which organic life can exist, but at which very many industrial processes are carried on, is constantly increasing. I venture to say that no method in learning how to investigate those properties will prove more fruitful of results than the method whereby phenomena are viewed in the light of their natural physical connections and the analogies which join them together in groups and classes.

Palmer Physical Laboratory,
Princeton, N. J.,
July 24, 1913.

DISCUSSION.

J. W. Richards: I would call attention to the comparison of heat flow and electric flow on pages 91 and 92, and, first of all, to No 14 (which I freely confess I cannot understand) that no "thermomotive force is recognized which acts upon heat to move it," which seems to me contradictory to statement No. 2 which is,

"The cause F of the flow of heat is difference of temperature," and "the cause of flow of electricity is the difference of potential."

Two points have struck me in experimenting on heat flow which emphasize the great differences between the measurement of heat resistance and electric resistance. The first is the fact that you are always losing heat through radiation through space and you are losing it from all over the surface of the body, whereas the electric flow is practically continuous from one end of the body to the other without this loss by radiation; this difference, I think, is the one which is the reason for Professor Northrup's bringing out these methods. In these important improvements for measuring heat flow there is a very great quantitative difference, not a difference in kind, but in degree, between heat flow and loss of heat by radiation, and electric flow and loss of electricity by leakage.

The second point I wish to emphasize in making measurement of heat conductivity is the relatively large capacity of the body whose conductivity is being measured to store up heat. An electric charge is imparted to a body in a fraction of a second, whereas the amount of heat required to bring the body to thermal equilibrium is very great and it takes minutes and sometimes hours to obtain that state.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Denver, Colorado, September 9, 1913, President E. F. Roeber in the Chair.

THE HEAT RESISTIVITY OF CARBON AND GRAPHITE

By Jos. W. Richards.

Messrs. J. P. Stokes and A. D. Jamieson have recently determined in this laboratory the relative heat resistivities of carbon and graphite in the following manner: Two blocks each 20 x 20 x 10 cm. (8 x 8 x 4 inches) were placed on each other so as to form a cube of 20 cm. (8 inches) on the side. In the center a cubical cavity was made 2.5 cm. (1 inch) on the side. Two passages 1 cm. (0.4 inch) in diameter were then made from the outside to the inner cavity, and a smaller hole 5 mm. (0.2 inch) in diameter was drilled from the middle of an outside face to within 1 mm. (0.04 inch) of the inner cavity. The larger passages were for the introduction of electric-light carbons, passing through glass tubes which fitted tightly into the passages; the small hole was for a thermo-couple (iron-nichrome) for measuring the temperatures when thermal equilibrium was reached. Currents up to 1,000 watts were used in the arc, and many precautions were taken to obtain accurate results, particular care being given to running the tests long enough to reach equilibrium.

In calculating the heat conductivity of the material, the cube was conceived of as split into six truncated square pyramids, corresponding to the six outer and inner sides of the cube and the internal cavity, each pyramid having therefore a square base 20 x 20 cm. (8 x 8 inches), a top surface 2.5 x 2.5 cm. (1 x 1 inch), and a vertical height, or distance between base and apex, of 8.75 cm. (3.5 inches). The mean conducting section was taken as $\sqrt{20^2 \times 2.5^2} = 50$ sq. cm. = the geometric mean of the inner and outer surfaces.

From several careful determinations the following figures for thermal resistivity (in thermal ohms, *i. e.*, degrees per watt) of these two materials were obtained:

National Carbon Co. carbon	(202°-853°)	= 36.8	thermal ohms for a cm. cube
Acheson graphite	(240°-420°)	= 3.69	" " " " " "
		Ratio = 10 : 1	

From a discussion of the possible errors it would appear that this ratio is a minimum, for the reason that, while the temperature over the whole outside surface of the graphite cube was sensibly uniform, the carbon cube was distinctly hotter outside in the middle of the faces, cooler in the middle of the edges, and coolest at the corners. But the outside temperatures recorded were those at the middle of the faces, from which it follows that the average temperature drop in this case, from inside to outside, was taken smaller that it really was, and that therefore the heat resistivity (degrees per watt) deduced therefrom is too low.

Practically the same values were obtained for graphite between 240°–270° and 370°–420°, and for carbon between 202°–540° and 272°–853°. One may conclude, therefore, that the ratio of the heat resistivities of carbon and graphite at temperatures below 1000° C. is at least 10:1. While the absolute figures 36.8 and 3.69 may not be accurate to within 20 percent, it is believed that the ratio is accurate to within 10 percent.

For purposes of comparison with other results that have been obtained there follow abstracts of the more important investigations.

FitzGerald and Forssell (These Transactions, **11**, 323-4 (1907)) point out the fact that in using similar carbon and graphite electrodes in an electric furnace the part of the graphite electrode outside the furnace attains a much higher temperature than the corresponding carbon electrode, due to the higher heat conductivity of the graphite. No actual figures for conductivity are given.

FitzGerald (These Transactions, **12**, 165-9 (1907)), by heating one end of similar bars of carbon and graphite and measuring the temperature of different points along the bars, arrives at a comparative ratio of 18.5:1.

Hansen (These Transactions, **15**, 295 (1909)) measures in a water jacket the heat carried out of an electric furnace by "idlers," *i. e.*, by electrodes not carrying current, and assigns the carbon: graphite ratio a value of 8:1.

Later (There Transactions, **16**, 344 (1909)), Hansen carries his experiments further and calculates absolute values for the heat conductivity of graphite and carbon. The value given for graphite is 1.65 watts per degree per inch cube, or, converting to resistivity, 1.54 thermal ohms per cm. cube; for carbon, 0.165 watt per degree per inch cube, or 15.4 thermal ohms per cm. cube (200°–3200° C.). Ratio, 10:1.

These results are further supplemented by an appendix (These Transactions, **16**, 350 (1909)) reporting tests made on rods heated at one end by a heating coil and cooled at the other by a water jacket, the intervening surface of the rod being insulated. Measurements of temperature along the rod, made with thermo-couples and thermometers, gave values for graphite ranging from 5.33 watts per degree per inch cube at 100° to 2.87 watts per degree per inch cube at 600°, or 0.48–0.87 thermal ohms per cm. cube, and irregular values for carbon ranging from 0.118 to 0.185 watts per degree per inch cube, or 13.7–19.2 thermal ohms per cm. cube.

Hering (These Transactions, **16**, 315, 317-28 (1909); **17**, 171 (1910); Proc. A. I. E. E., **39**, 304 (1910); Met. and Chem. Eng., **9**, 653 (1911)) passes a current through an electrode surrounded by an insulator and determines the conductivity, assuming the C^2R heat generated in the electrode to be lost entirely through the two ends of the electrode, measuring the temperature at the middle of the electrode by means of a pyrometer, and cooling the two ends by means of water jackets. The value obtained for graphite is 0.740 cal. per second per degree per inch cube; and for carbon 0.329 cal., or 0.82 thermal ohms and 1.9 thermal ohms per cm. cube at 900° C. Ratio carbon to graphite, 2.3:1.

Wologdine and Queneau (Electrochem. and Met. Ind., **7**, 383, 433 (1909)) placed a flat plate of the materials to be tested over a gas flame, measured the temperature of the two sides of the plate with thermo-couples, and measured the heat conducted through the plate by means of a continuous-flow water calorimeter. Queneau (These Transactions, **17**, 211 (1910)) gives values for

graphite and carbon obtained in this manner, 0.79 thermal ohm for graphite and 2.33 thermal ohms per cm. cube for carbon, up to 600° C. Ratio carbon to graphite, 3:1.

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DISCUSSION.

F. A. LIDBURY: I would like to ask for some further details as to how the temperature of the inner and outer surfaces was measured. It is stated that a small hole was drilled from the outside face to within one mm. of the inner cavity and this hole was for a thermocouple. What was the size of the wires that constituted the thermocouple and how were those wires insulated? Were experiments made to determine whether a different reading was obtained by the use of wires of a different gauge from those with which the experiments were actually made?

J. W. RICHARDS: I should say, the wires were one-tenth of a millimeter, about as large as a horsehair, and they were placed down in a small glass tube which fitted the hole closely. One was inside the tube and one outside with the junction at the end; the tube was wound around with some asbestos thread, so as to make a little plunger and prevent the circulation of air. This was carefully calibrated and also compared with our platinum thermocouple. We did not try different sizes of wires. I think the measurements of the temperature were reliable within five degrees of the correct temperature.

F. A. LIDBURY: It is always a matter of considerable doubt as to how nearly an arrangement of that nature gives you a temperature corresponding to the point where the thermocouple is placed; and it is very necessary in giving measurements to state exactly how the temperature reading was obtained.

C. A. HANSEN: Dr. Richards claims an accuracy of a degree or so at 800° C. for his thermocouple. We are continually buying such instruments and, if I remember correctly, the P. T. R. calibration sheets claim an accuracy of 10° at 1,000° C. This applies to ordinarily decent methods of determination. There is little question that atomic weight work accuracy will enable our own Bureau of Standards for instance to check the Physikalische Reichsanstalt closer than 10° C. at 1,000° C.

J. W. RICHARDS: I have some pure copper and I usually calibrate my own thermocouples at 1086°, and I find by doing that, if the couple has not been damaged, you can get within five degrees at that temperature. So I think that these temperatures varying from 400° to 800° are reliable.

C. A. HANSEN: I believe the copper points are used at the P. T. R. as calibration points.

J. W. RICHARDS: We know the melting point of copper to better than 10° C.

C. A. HANSEN: Dr. Richards has quoted my ratio of 8 or 10 to 1 for the heat conductivities of graphite and carbon. My measurements referred to an upper temperature of something like 3,000° C. and a lower temperature of less than 100° C. Since the carbon electrode material is almost completely identical with the graphite electrode material at the upper temperature the two must have somewhere nearly the same heat conductivity constants. When I obtained a ratio of 8 or 10 to 1 then for the entire temperature range it must be that at the lower temperatures the ratio is greater than 8 or 10 to 1. Personally, I think that FitzGerald's old ratio of 20 to 1 for the low temperature range comes pretty near to being correct.

J. W. RICHARDS: I wish some one who has thought over the matter could give us some inkling why Wolgodine and Queneau obtained figures down to the ratio of 3 to 1.

CARL HERING (*Communicated*):[1] When one determines a physical constant and finds the value to differ greatly from those found by others, it seems to me one ought to carefully compare the conditions under which the determinations were made to see

[1] Read by C. G. Schluederberg.

whether the values really do represent the same physical quantity, and, if not, to call attention to this difference, in justice to both values.

Moreover in case there are such large differences, and especially in papers before this Society, sufficient details of the original measured data, as distinguished from the mere final results calculated from them, should in my opinion be given, so that others can form their opinion of the probable correctness of the final result.

Although graphite obtained in the electric furnace may perhaps be considered an elemental quantity, like one of the elements, and therefore be assumed to have nearly constant physical properties, yet carbon can surely not be so considered, as it is no doubt made differently intentionally by the makers to adapt it better for specific purposes. Hence close agreement in the physical constants of carbon can hardly be expected.

Prof. Richards' paper is entirely devoid of any details which would enable others to form their own opinion of the probable reliability of his results. The statement "current up to 1,000 watts" (*sic*), makes matters still worse. What was the current in amperes? Why not state it? The carbon electrodes for the arc must have been very small, and if he means by the peculiar statement just quoted, that a kilowatt of heat was continuously being set free in the interior, the current was presumably very great relatively to those small electrodes, and must therefore have developed much heat in them. The loss of heat through these electrodes follows an entirely different law than that through the walls. The amount lost through them must moreover have been relatively quite different in the carbon block than in the one of graphite. The temperature gradient must have been quite different in the two walls through which these very hot electrodes passed, than in the other four.

It must have taken many hours to reach the real "stable state" and unless this state was really reached the results are worse than worthless. Moreover during that long time of many hours, much carbon must have been burned away from these very thin electrodes in a kilowatt arc; how much heat did that add—and was it allowed for?

Assuming the temperature of the arc to have been 3,500°, the heat gradient in that 1 mm. wall between the arc and the pyrometer was at the rate of about 77,000° C. per inch for the graphite and about 66,000 for the carbon. As this is unbelievable, there must be something radically wrong in the measurements, hence also in the results based on them. The metal thermocouple probably chilled this thin wall, hence did not take the true temperature. If in the calculations the temperatures at that point 1 mm. from the arc vapors had been taken to be nearly 3,000°, which they probably really were in the other five walls, instead of 420° and 853°, his calculated resistivities would have been only a small fraction of the values which he deduced, hence radically different.

His statement that if the individual figures of a quotient are accurate to within 20 percent their ratio is accurate to 10 percent, is not borne out by simple arithmetic. The ratio may then be incorrect as much as 50 percent instead of 10 percent. Did he obtain just one set of readings, or if more, how did they agree among themselves?

The curve of the temperature gradient in the thick wall of a cube is quite different from that in a thin slab, a rod, or a current-carrying electrode; hence his value (which is a mean for the particular heat gradient) would necessarily be different from those obtained by the others, which he gives "for comparison."

In this respect his results are not at all comparable with those from my electrode tests which he quotes. The heat gradients are entirely different in the two cases, and in the electrodes the heat is generated in the heat conductor itself, and each calorie has a different length of path to travel to get out. My results were obtained with great care and the measurements were made very carefully with accurate instruments (potentiometer) and were the averages of many closely agreeing readings; and the method did not involve any serious inherent errors. Hence, I believe them to be accurate and reliable, so far as they went.

This determination by Professor Richards was a repetition of a somewhat similar one made in his laboratory by Mr. Kemmer (though not quite as I suggested it to him two years previously), and was described by Prof. Landis in these Transactions (Vol. XVIII. p. 224, (1910)). The values then obtained, when reduced

to the now generally accepted unit, thermal ohm, were about 57 to 11 for carbon, and from 2.8 to 0.11 for graphite. My discussion of those earlier tests, p. 229 of that volume, would nearly fit the present paper also.

J. W. RICHARDS: Dr. Hering starts out by saying that his results are not comparable with mine because they were made under a different condition; that is, that he determined what he calls the electrode mean, and he says I should have stated this, so that people would not be misled. If the results are not comparable, I hardly see that Dr. Hering has any ground for saying that my results are wrong because they differ from his.

Dr. Hering has a curious idea of what happens in the center of a small cube of this sort. If we drill a hole within 1 mm. of the inside of the surface and have the inside heated by an arc Dr. Hering believes, apparently, that the inside face of this cube is at the temperature of the arc; therefore, that there is a drop of some 3,000 degrees in the first millimeter. This is evidently a misconception of the whole idea of heat flow from an inside to an outside surface, because no matter what the temperature of the source of the heat is, the inside cannot get hotter than is necessary to transmit the amount of heat which is passing out from the inside surface to the outside surfaces. The temperature of the inside surfaces may be 300°, 400° or 500°, whereas the source of the heat may be the arc at 3,700°. It is the same old story of the gases inside a boiler being hotter than the water tube of the boiler, so that Dr. Hering's criticism on that last millimeter is a fundamental misconception on the face of it.

Many of Dr. Hering's criticisms would have been obviated by my giving the detailed data, but I wish to call attention to the fact that the electrodes which were used were small carbon electrodes. While the energy was 1,000 watts, the amperes varied about 30, and the volts about 35. No one appreciates better than myself how long it takes for equilibrium to take place in these cubes; we would start them early in the morning and in the afternoon we would make the measurements to see if it had attained equilibrium; the corresponding time between graphite and carbon is very different.

CARL HERING (*Communicated*): Prof. Richards cannot find

in my discussion the statement which he says I made, namely that his results were wrong *because* they differed from mine. I claimed that his results were unreliable regardless of what mine were; and I did say that the two were not directly comparable as they are different means, just as the arithmetic and geometric means are different. Prof Richards' reply to my remarks about the 1 mm. wall, merely strengthens my original opinion about his determination. I have nothing to retract.

J. W. Richards (*Communicated*): Dr. Hering's amazing statement that he has "nothing to retract" concerning that last millimeter, even after hearing my reply to his criticism, calls for further reply. If a scientist ordinarily so keen and clear in his reasoning as Dr. Hering is still unconvinced about that last millimeter, then it is probable that others may have their doubts, and it becomes necessary to explain in greater detail the fundamental misconception in Dr. Hering's reasoning.

Take a hollow sphere or cube and assume a source of heat in its interior, generating a certain number of watts or calories per second. When equilibrium of temperature conditions is finally attained, the outside of said sphere or cube will have attained that fixed temperature at which it can disperse the steady heat flow coming from the source within. Also, at the same time, the inner surface of said hollow sphere or cube will have attained that fixed temperature necessary to transmit through the walls the steady heat flow in question; this inner surface temperature is conditioned only by the outside surface temperature, the thickness of the walls, their heat conductivity, and the amount of the heat flow—it is entirely independent of the temperature of the source of heat within the inner cavity. Or, in other words, given a hollow sphere with a certain outside temperature, dispersing a steady heat flow from its interior, and given the thickness of the walls and their heat conductivity, the *necessary* temperature of the inside surface is fixed by these conditions, and is entirely independent of the temperature of the source of heat inside. It is exactly upon this principle that the roof of an open-hearth furnace does not melt, although the temperature of the hot gases in the furnace is frequently 1,800° to 1,900° C., and yet the melting point of the roof bricks is only 1,700° C.; the fact that heat

is flowing through the bricks keeps down the temperature of their inner surfaces, and prevents them from melting. This principle is so fundamental, and its proper appreciation and utilization so important in all applications of high temperatures, that I commend a careful study of the matter to every technologist who has any lingering doubts on the question.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Boulder, Colo., September 10, 1913, President E. F. Roeber in the Chair.

POSSIBLE APPLICATIONS OF THE ELECTRIC FURNACE TO WESTERN METALLURGY

By Dorsey A. Lyon and Robert M. Keeney.

INTRODUCTION.

In *Metallurgical and Chemical Engineering* for March, 1912, there appeared a very interesting editorial upon the statement of economists that value is created when any artificial product is turned into some form useful to the community; likewise, that value is of three kinds, namely, "time value," "place value" and "form value." To illustrate this point, the water of the Grand River at Shoshone had little value before the construction of the plant of the Central Colorado Power Co.; converted into electric energy at Shoshone it has form value, and, transmitted to Denver, place value. So it is with a large number of ore deposits of the Western parts of the United States. Their location is such that at the present time they have no more value than does the water at Shoshone. Such being the case, is it possible to so treat these ores that their valuable constituents may be given a form value which will enable them to command a place value, and, if so, can the electric furnace be used to advantage in doing so?

As has been repeatedly pointed out by the writers whenever the opportunity has presented itself, the electric furnace was not developed for the purpose of competing with the combustion furnace in the domain of ordinary metallurgy, but rather to do for metallurgy what it is not possible to do with the combustion furnace, either because of existing local conditions, or because a higher temperature or method of heating is required than it is possible to obtain in the combustion furnace.

In this paper we will discuss only those applications of the electric furnace which are strictly within the domain of metal-

[1] Published by permission of the Director of the U. S. Bureau of Mines.

lurgy, and where the electric current is used as a thermal agent only. Also, we will discuss the use of the electric furnace only in so far as it has to do with the smelting of ores, having in mind especially those conditions which are to be found in the Western part of the United States.

With the exception of the production of aluminium (which will not be discussed in this paper), iron, and ferro-alloys, electric furnace metallurgy is largely in the speculative or experimental stage at the present time. In that which follows we will endeavor to briefly outline the present status of the electric furnace in the smelting of ores, and likewise the production of ferro-alloys.

PRESENT STATUS OF THE ELECTRIC FURNACE IN SMELTING IRON ORES.

As is well known, of the coke charged into an iron blast furnace only about two-thirds of the same is used for producing the heat necessary for carrying on the process, while the other one-third is used as a reducing agent. Therefore, if the electric furnace is used for the smelting of iron ores, only enough carbon has to be supplied to unite with the oxygen of the ore, in other words, to reduce it, and for this reason the smelting of iron ores in the electric furnace is of much importance to the Western Coast States, for there we find comparatively large iron ore deposits, but not suitable blast furnace coking coals, and so the cost of coke makes ordinary blast furnace smelting prohibitive. However, it is, of course, necessary to supply carbon in some form as a reducing agent, and, even though only one-third as much carbon is required for this purpose as is required in ordinary blast furnace practice, it may be impossible to obtain even this amount at such a cost as will permit of the use of the electric furnace.

Moreover, so far only coke and charcoal have been tried to any extent as reducing agents in the reduction of iron ores in the electric furnace, and of these only charcoal has proven satisfactory. This has been shown by repeated trial runs at Trollhättan, and has been forcibly demonstrated by the fact that the plant at Hardanger, Norway, where coke was used as a reducing agent, after being in operation for about nine months was forced to close down, due to the fact that nowhere near the same amount of pig iron could be produced per kilowatt year of electrical

energy expended as can be produced when charcoal is used. That this is true is doubtless due to the fact that coke is a much better conductor of electricity than is charcoal, especially after it becomes hot. For this reason, when using coke the resistance of the charge becomes lowered, and, as the smelting in the electric iron reduction furnace is done by the heat produced by the resistance which the electric current meets with in passing through the charge between the electrodes, more electrical current (amperes) is required to produce the same amount of heat. Likewise, as stated by Crawford,[2] the electrical conductivity of coke "is so good that much of the current passes between the electrodes in the upper part of the furnace. The smelting zone is thereby raised, and the furnace runs hot on top, with attendant melting of the arches (of the roof of the crucible), and cold at the bottom. Further, the coke, because of its density and high crushing strain, does not break down like charcoal as the burden descends, hence less surfaces of carbon are exposed to be oxidized by the ore, and there is a less intimate mixture of the two.[3] Reduction of the ore takes place more slowly, the silicon in the iron is lowered, the consumption per ton increases, and the efficiency of the furnace is reduced."

Inasmuch as either charcoal or coke are the only practical reducing agents we know of at the present time, that is, for use on a large scale, and as it would seem from what has just been stated that it is impractical to use coke, this necessarily limits the electric iron reduction furnace to the use of charcoal. Hence it is necessary that such a furnace be located in close proximity to a well timbered region, where charcoal as well as electric power can be produced at a low cost. If this be true (and it undoubtedly is at the present time), it is unfortunate, especially as regards the situation in southern California and in other of our Western States, where there are quite large iron deposits of A-No. 1 ore, but where coke is too expensive to permit of ordinary blast furnace smelting, and where the use of charcoal for electric furnace work is entirely out of the question, as there are no forests to furnish the wood necessary for its production. On the other

[2] Progress of Electric Smelting at Héroult, California, by John Crawford. Metallurgical and Chemical Engineering, July, 1913, p. 386.

[3] This intimate mixture is, of course, very necessary where the reduction is almost entirely performed by solid carbon, as is the case in the practice at Héroult, California. —THE AUTHORS.

hand, crude oil is generally more or less plentiful and comparatively cheap in such districts, especially in California. Aside from its possible use in connection with electric furnace work, those interested in the subject have for years been considering the possibility of using crude oil as a reducing agent. So far as we are aware, this has not as yet been successfully done. That the carbon and the hydrogen of the oil will reduce iron oxides is self-evident, but as yet no one seems to have been able to solve the problem as to how to bring the ore and the oil together at the proper temperature. As a result of a preliminary investigation conducted by the metallurgical section of the U. S. Bureau of Mines on the possibility of using crude oil as a reducing agent, it seems as if about the only manner in which crude oil may be used for this purpose is to first convert it into a fixed gas and then introduce this into the crucible of an electric furnace, thus preheating the gas to such a temperature that it will effectively reduce the iron oxides as it passes up through the shaft of the furnace. Be that as it may, it is to be sincerely hoped that ultimately a suitable process may be devised whereby oil may be used as a reducing agent, and thus broaden the field for the possible application of the electric furnace in the reduction of iron ores; for, from what has been stated above, it is apparent that the electric furnace is limited for such purposes at the present time to those localities where charcoal and electric power are comparatively cheap. As cheap power is a prerequisite not only in the smelting of iron ores, but of all other ores as well, which it is proposed to treat in the electric furnace, the cost of power will be discussed later.

That the electric furnace has been successful in the smelting of iron ores in those districts which are favorable to the same is shown by the fact that ten furnaces of the Swedish type (a total capacity of about 22,000 kilowatts) have been erected in Sweden, Norway and Switzerland. In this country there is one electric furnace iron plant of two furnaces, with a total capacity of 5,000 kilowatts, in operation at Héroult, California. The type of furnace used in California is different from that used in Sweden, due to the fact that a different grade of iron is desired (and can be produced in the California furnace) from that which is produced in Sweden; that is, in Sweden a metal is produced which

is low in silicon and carbon and which is particularly well suited to steel making, whereas in California the demand is for a soft grey foundry iron.

As to the quality of pig iron produced in general in the electric furnace, the engineers in charge of the experimental work at Trollhättan made the following statement in their final report:

"*The Quality of the Pig Iron:*—The silicon content does not vary more than in an ordinary blast furnace. The phosphorus content is lower than with the same quality of charge in an ordinary blast furnace; this is due to the lower consumption of charcoal. The percentage of sulphur is, however, slightly higher in the electric furnace. But it should be observed that both at Trollhättan and at Hagfors unroasted ores have been used without any difficulty arising from the sulphur present."

The quality of the electric pig iron has been highly commended. It acts particularly well in the open-hearth furnace, and steel made from it is certainly not inferior to steel made from ordinary pig iron. Mr. E. Odelberg, managing director of the Stromsnas Iron Works, states that the electric pig iron is "of the very best quality for the open-hearth process, and, as regards the uniformity of the silicon content, fully as uniform as iron from an ordinary blast furnace." Mr. A. Herlienus, managing director of the Uddeholm Company, states that "the electric pig iron has been used with satisfactory results both for the open-hearth, Bessemer and the Lancashire processes. Generally speaking, there has been no difficulty in obtaining pig iron of uniform quality, although slightly better uniformity may possibly be obtained with a very carefully conducted blast furnace."

"*Use of Concentrates:*—It has been found that the proportion of concentrates ought not to exceed 20 per cent. of the ore charged. This figure, however, does not appear to be final, as in a later constructed and somewhat modified furnace of the same type at the Hagfors Iron Works, 25 percent concentrates are used without any difficulty.

"*Power Consumption:*—The power consumption per ton of pig iron varies in proportion to the iron content in the ore. A poor ore and pig iron high in silicon and manganese require more power than a rich ore and pig iron low in silicon and

manganese. For such iron the power consumption averages only 2,067 kw. hours per ton of pig iron; *i. e.,* there is obtained 4.22 tons of pig iron per kw. year, or 3.10 tons per h.p. year.

"*Charcoal Consumption:*—The charcoal consumption per ton of pig iron varies from 20 to 24 hectolitres (56 to 68 bushels), depending on the quality of the charcoal and the charge. Coke has been found to be unsuitable for this furnace unless mixed with charcoal.

"*Consumption of Electrodes:*—The consumption of electrodes at Trollhättan has been reduced to less than 3 kg. (6.6 lb.) per ton of pig iron. At Hagfors it has amounted to as much as 6 to 9 kg. (13.2 to 19.8 lb.). This discrepancy is explained by the fact that the electrode consumption is increased in proportion to the higher power consumption for a poorer charge, and is further increased by the more efficient circulation of gases and higher CO_2 content in the gas. The lower electric load per unit of surface at the Hagfors furnaces also contributes to the higher electrode consumption at this plant.

"*Cost of Repairs:*—The cost of repairs is lower than there appeared at first to be reason to expect. In the manufacture of pig iron containing silicon and manganese the costs for repairs are higher than in producing pig iron with low contents of those elements.

"*Value of the Gas:*—At the plant of the Uddeholm Company, at Hagfors, the gas from the furnaces has been used with very good results for heating the open-hearth furnaces. It is estimated that the value of the gas obtained per ton of pig iron may be taken at 2.50 kroner (about 67 cents)."

We quote from the report a summary of the most important figures relating to the economical results. These show that step by step the results have been improved, the quantity of iron per h.p. year increased, while the electrode consumption and time for repairs have been reduced, these three items, in conjunction with the saving in charcoal, being the decisive factors as regards electric iron smelting. See Table I.

The figures show that during the last few months as much as

3.20 to 3.10 tons of iron have been obtained per h.p. year, while before the alterations then made the highest average figure was 2.86 tons. The highest expected yield was 3 tons per h.p. year, which figure has thus been exceeded. It may also be mentioned that during single periods of several weeks while especially suitable ores were used, the highest average figures above mentioned have been very materially exceeded. It will thus be seen that, as regards efficiency, the results of the furnace have surpassed expectations. The electrode consumption was 13.8 kg. (30.4 lb.), which has finally been reduced to about 3 kg. (6.6 lb.) per ton of pig iron. The cost and time required for repairs were items which could not be estimated beforehand. Experience has shown that both the cost and the time required are less than could have been hoped for. Wherever employment for the gas can be found, this should also be taken into account, as is being done at Hagfors, where its value for firing the open-hearth furnaces is estimated to reduce the cost of the pig iron by about 66 cents.

TABLE I.

	Nov. 15th, 1910, to May 29th, 1911	Aug. 4th, 1911, to June 21st, 1912	Aug. 12th, 1912, to Sept. 30th, 1912	October to December 1912
Ore, concentrates and briquettes..kg.	4,336,338	7,917,214	1,406,530	2,914,830
Limestone .....kg.	345,405	647,479	108,150	169,944
Charcoal .....hl.	65,474.5	107,282.5	21,859.5	44,934.5
Coke .....kg.		70,854		
Electric energy .....kw.-hrs.	6,339,131	10,845,180	1,939,073	3,957,565
Iron content of ore.....percent	60.79	60.75	68.67	65.38
Iron produced .....kg.	2,636,098	4,809,670	965,915	1,905,865
Slag per ton of iron.....kg.	350	324	192	
Electrodes per ton of iron..gross kg.	10.00	6.08	3.02	2.78
Electrodes per ton of iron...net kg.	4.95	5.17	3.02	2.78
Charcoal per ton of iron.....hl.	24.84	22.31	22.63	23.58
	Hr. Min.	Hr. Min.	Hr. Min.	Hr. Min.
Working time	4,441 20	7,218 23	1,173 08	2,158 30
Repairs	236 53	506 07	13 47	49 30
Repairs in percent of total time....	5.06	6.55	1.16	2.24
Average load .....kw.	1,427	1,502	1,653	1,833
Kw.-hrs. per ton of iron	2.405	2.255	2.007	2.076
Iron per kw.-year .....tons	3.64	3.88	4.36	4.22
Iron per h. p. year.....tons	2.68	2.86	3.20	3.10

POSSIBILITY OF SMELTING COPPER ORES IN THE ELECTRIC FURNACE.

So far as we are aware, no copper ores are being treated at the present time in the electric furnace in this country. It is reported, however, that in Norway trial smeltings of copper ores with an electric furnace of 1,000 h.p. and an estimated producing capacity of 2,000 tons per annum have been conducted at the Ilen Smelting Works, Trondhjem, and we understand that it is the intention to smelt copper ores regularly at this plant in the electric furnace.

More or less experimental work has been done upon the subject, and, as a result of this work, and reasoning by analogy, there seems no good reason why copper-bearing ores cannot be as successfully treated in an electric furnace as in a combustion furnace. In all furnaces of the latter class which are used for this treatment of copper ores the fuel used takes no part in the reactions which are necessary for obtaining the desired product, unless it be in the reduction of oxide ores which are smelted alone, that is, without an admixture of sulphides, which is practically an unheard of thing in this country at the present time. For example, in the reverberatory furnace the fuel acts only as a heating agent; in blast furnace smelting, if what is known as ordinary blast furnace smelting is used, the coke added to the charge is for the purpose of supplying the heat necessary to raise the charge to such a temperature as will permit of the necessary reactions between the oxides, sulphates and sulphides present, and to scorify the resultant mass and thus permit of the separation of the slag and matte. In semi-pyritic and pyritic smelting the necessary oxidation of the sulphides and iron is brought about by the oxygen of the air entering the tuyeres, and the coke used is simply for the purpose of supplying the amount of heat necessary for the successful carrying out of the process, which is not supplied by the oxidation of the sulphur and the iron present in the charge at the time it passes through the tuyere zone of the furnace. Such being the case, there seems to be no reason why the smelting of copper ores could not be done as well by electric heat as by that derived from the combustion of coke, especially if local conditions warrant it. The writers, as a result of experimental work which they have done in connection with tests which have been made by the U. S. Bureau of Mines, and

likewise judging from results which have been obtained by others, believe that it is perfectly feasible, both metallurgically and commercially (the latter, of course, depending upon local condition) to use the electric furnace for the smelting of copper ores.[4]

LEAD ORES.

The smelting of straight lead ores in the electric furnace has never been attempted either commercially or on a large experimental scale, largely because of the ease and cheapness of smelting by combustion processes. For the smelting of ordinary lead ores, it has no especial application, but in the treatment of complex lead-zinc-silver ores the electric furnace might be profitably used. The roasted ore could be treated in an electric furnace operated at a temperature such that the lead reduced by carbon melts and collects the precious metal values and the zinc is volatilized and condensed.

ELECTRIC ZINC SMELTING.

The use of the electric furnace in the metallurgy of non-ferrous metals has had greater application for the treatment of zinc ores than in the metallurgy of any other of the non-ferrous metals except aluminium and ferro-alloy manufacture. Since 1885, when an electric furnace for the treatment of zinc ores was patented by the Cowles Bros., experimental work has been conducted on the subject. The commercial application of the process, however, has not resulted to any great extent because of the difficulty of condensing the zinc vapor produced under the smelting conditions of the electric furnace. The cause of this difficulty has not yet been definitely determined. With few exceptions the experimental work has not been conducted on a very large scale, and so it may be said that the electric smelting of zinc ores is still in the experimental stage.

Undoubtedly the electric furnace has a very promising field in the metallurgy of zinc, but before its wide application can be expected the condensation problem must be solved and mechanical problems in connection with the electric furnace worked out. The

[4] Those desiring a more complete and extended discussion of this subject are referred to a paper by the writers, entitled "Smelting of Copper Ores in the Electric Furnace," which was presented at the August, 1913, meeting of the American Institute of Mining Engineers, at Butte.

latter should not be difficult. The special field for electric furnace work in zinc metallurgy is due largely to the low thermal efficiency of the present zinc retort which is given by Richards[5] as only 7 percent.

With the efficiency of the electric furnace varying from 50 to 75 percent, the difference in the heat necessary for carrying out the smelting operations is so great as to permit of a higher cost of electrical energy than is permissible in many electro-metallurgical operations.

PRESENT METHODS OF ZINC SMELTING.

The present method of zinc smelting consists briefly of the roasting of sphalerite, ZnS, to zinc oxide, ZnO. The roasted ore is then smelted with excess of carbon as reducing agent, in a closed fire-clay retort having a condenser attached to it. The zinc oxide is reduced to zinc at a temperature of 1033°C. and, as the boiling point of zinc is given by various authorities at from 920 to 930° C., the reduced zinc passes off as a vapor and is condensed as liquid metal in a condenser. In order to make the reaction more rapid, the temperature of the retort walls is kept at from 1000 to 1300°C.

Zinc vapor condenses into liquid in the range of temperature from 420°C. to 860°C. The lower temperature is that of solidification of zinc, and the upper approaches the vaporization point. In practice it is the aim to keep the temperature at 500° C., so that the zinc can be tapped off molten from the retort. If the temperature of the condenser is too low, the vapor too dilute, or if the vapor is diluted with some carbon dioxide instead of carbon monoxide or with metallic vapors other than zinc, there will be a formation of blue powder, which consists of minute particles of zinc, each one apparently covered by a thin coating of zinc oxide. Because of this coating of oxide the powder cannot be converted to spelter by a simple melting.

The process is intermittent, and the retort holds only about 200 pounds of the mixture. A charge is smelted, the zinc removed from the condenser and the residue raked out of the retort. Because of the small scale and intermittent nature of the operation, the labor cost is high. Likewise the thermal efficiency is low

[5] J. W. Richards, Metallurgical Calculations, Part 1, page 80.

because of the heat being conducted to the charge through the furnace walls. The retorts form an expensive part of the process, for at the high temperature necessary they last only about a month.

ELECTRIC FURNACE METHOD OF ZINC SMELTING.

Processes for the electric smelting of zinc ores are of two general classes—first, reduction of zinc oxide and its compounds by carbon and carbon monoxide, and second, decomposition of zinc sulphide by metallic iron. Examples of the first method are the DeLaval and Johnson processes, and of the latter, the Cote-Pierron and Imbert-Thomson-Fitzgerald processes. Reduction of zinc oxide with carbon is based upon the reactions

$$(1)\ ZnO + C = Zn + CO$$
$$(2)\ ZnO + CO = Zn + CO_2$$
$$(3)\ CO_2 + C = 2CO$$

The use of iron is according to the reaction

$$(4)\ ZnS + Fe = Zn + FeS$$

In neither of these processes does the electric current perform any other function than heating. Due to the fact that alternating current is supplied to the furnace, there is no electrolysis. As will be shown later, the process may be performed in either an arc or resistance furnace.

One essential difference between the reduction of zinc oxide by carbon in an electric furnace and in a retort is that the retort process is intermittent and the electric process may be continuous. In the electric furnace the charge is added at intervals, as necessary, and the slag is tapped or allowed to run continuously from the furnace without disturbing the operation of the process.. The reduction by carbon or carbon monoxide occurs just as in the retort, with the exception that because of the rapidity of reduction in the present designs of electric furnaces, the third reaction does not take place apparently as well in the electric furnace as in the retort, resulting in a greater amount of carbon dioxide in the zinc vapor, which has a bad effect on the condensation. This results in the production of a large amount of blue powder.

Various attempts have been made to reduce this, the principal one being the passing of the vapor through incandescent carbon before entering the condenser.

ZINC SMELTING AT TROLLHAETTAN, SWEDEN.

The largest plant in commercial operation for the electric smelting of zinc ores is that of the Norse Power and Smelting Syndicate at Trollhättan, Sweden, which also has smaller works at Sarpsburg, Norway.

The Trollhättan plant has a furnace house 300 by 52 feet, which contains 11 resistance furnaces, six on one side of the building and five on the other, two refining furnaces and several arc furnaces. In 1912 it was reported that the new furnace house, then partially erected was to contain eight furnaces of from 600 to 900 kilowatts each. The old furnaces are of 270 kilowatts each. The total power now being used at the works is 3,000 kilowatts, and with the new installation it will be 7,500 to 10,000 kilowatts.

The Sarpsborg plant has three arc furnaces and four refining furnaces.

For the last six or seven years zinc smelting has been conducted at these works in the DeLaval furnace, but part of the product has been the result of refining dross and scrap. It is stated that now ore smelting, using carbon as a reducing agent, is being performed. The only information available about the works is that contained in a report of F. W. Harbord,[6] published in 1911, as everything in regard to the process and the results obtained is kept as secret as possible.

The smelting process consists essentially of two operations, namely, concentration of the zinc to a rich oxide followed by reduction in another furnace. Oxide ore, flux, and carbon used as a reducing material, are charged into the furnace, where most of the zinc and some of the lead are volatilized and condensed partly as metal and partly as blue powder and oxide, containing 54 percent zinc and 20 percent lead. This is really a concentration powder, for the blue powder is now mixed with ore and recharged, when a larger percentage of the metal volatilized is recovered as metal. The remainder of the lead, which carries some silver, is reduced to metal in the smelting hearth

[6] Engineering and Mining Journal, February 10, 1912, p. 314.

and is tapped with the slag. Some lead, zinc and silver passes into a matte and some into the slag.

The furnaces are of a modified Siemens type with a shallow hearth, and covered with a roof. A carbon block bottom forms one electrode while the other passes vertically through the roof.

In some furnaces the ore is charged through the roof, in others at one side of the electrode, while others have been designed having a continuous side feed.

The consumption of electric current in the experimental run at Trollhättan averaged 2078 kilowatt hours per ton of ore smelted, which does not include re-smelting the powder. Two tons of powder were re-treated per ton of ore smelted.[7] A comparison of the resistance furnaces at Trollhättan with the arc furnaces at Sarpsburg shows that while one was as good as the other for the carrying out of the desired reaction, the energy consumption was 70 percent more in the arc furnace than in the resistance furnace. The consumption of electrodes was also higher in the arc than in the resistance furnace, being 89.2 pounds per ton of ore at Sarpsburg and 69.3 pounds per ton at Trollhättan.

Harbord states: "The weak point of the process is the large amount of metallic powder produced in proportion to the metals. The recovery of a large percentage of metals is admittedly a very difficult problem, but that present practice can be very greatly improved upon I have not the least doubt, leading to decreased consumption of energy and reduction of labor costs. One detail which is a great improvement on the early practice is the better separation of the crude zinc from the powder by a mechanical stirrer."

At the time Harbord made his report, Mr. J. C. Moulden,[8] of the Sulphide Corporation, which ships the Broken Hill slimes, which are used at Trollhättan, stated he was of the opinion that the extraction, when certain improvements were made, would eventually be 75 percent of the zinc, 80 percent of the lead, and 80 percent of the silver.

The cost of power at Trollhättan is about $10 per kilowatt year, or 0.11 cent per kilowatt hour.

[7] Engineering and Mining Journal, July 6, 1912, p. 7.
[8] Metallurgical and Chemical Engineering, 4, 1911, p. 673. Mining Magazine (Lond.), October, 1911.

EXPERIMENTS AT MC GILL UNIVERSITY.

For several years extensive experimental work on a laboratory scale has been conducted under the direction of Dr. Alfred Stansfield and W. R. Ingalls.[9]

This experimental work has been largely directed toward a study of the condensation problem and the speed of reduction. Uncommonly high percentages of carbon dioxide were found in the gases from the electric furnace, which, as previously stated, prevents condensation of the zinc as metal by oxidizing the condensing globules of the latter and preventing coalescence. This has been noted by other experimenters, as well as the presence of considerable quantities of carbon and ore dust in the condenser, the latter believed to be due to stirring up of dust in feeding the charge into the furnace.

Experiments were also conducted on the reduction of zinc oxide in an intermittently heated retort. The charge was introduced into the retort at the beginning of the operation and at the end of the same the residue was discharged as an unfused residue. Reduction took place very slowly and the results obtained showed that while it might be possible to operate the process continuously such a course was not feasible. When the furnace was started with a bath of slag and the charge introduced continuously upon the bath, the reduction became relatively rapid. No explanations were given for this, but Ingalls states that a retort of small proportions smelts a surprisingly large quantity of charge in comparison with the retort of ordinary practice at an equivalent temperature. With a furnace consisting of a plumbago crucible 12 inches (30 cm.) in diameter, 10 pounds (4.5 kg.) of ore per hour was smelted. With a furnace 18 x 18 inches (45 x 45 cm.) in horizontal section, they smelted 737 lbs. (335 kg.) in 23 hours.

The statements as published are not complete, however, for the output of an electric furnace is due chiefly to the energy input and not to the interior volume of the furnace. The capacity in kilowatts of the furnaces used is not given, but it is clear that in comparison with a combustion retort the electric furnace has a high output per unit of interior volume.

[9] W. R. Ingalls, Engineering and Mining Journal, July 6, 1912, p. 7.

As the electric furnace may have a high temperature it is possible to drive off most of the zinc, leaving a low zinc slag to be tapped from the furnace. A calcareous slag as well as a high temperature is also necessary. At McGill University, the slags contained less than one percent of zinc.

Interesting figures of power consumption were obtained in the experiments at McGill. In one run lasting 24 hours, with a resistance furnace 18 inches by 18 inches (45 x 45 cm.), the power consumption was 1610 kilowatt hours per short ton of ore smelted. Theoretically it is estimated that from 955 to 1060 kilowatt hours per short ton of ore are necessary to smelt ordinary zinc ore. Ingalls concluded that a figure of 1200 kilowatt hours per short ton would put electric smelting upon a sound basis so far as power consumption is concerned. He also somewhat pessimistically concluded that there is some chance for electric zinc smelting if it becomes possible to smelt with 1200 kilowatt hours per ton of ore, to reduce materially the labor and electrode costs, and to extract 90 percent of the metal.

Previous to the experiments of Stansfield and Ingalls, Stansfield and Reynolds conducted experiments on the smelting of lead and zinc sulphide ores carrying silver, with the intention of distilling and condensing the zinc, of reducing the lead in the molten form and of collecting the silver with the lead. These runs showed the possibility of smelting these ores in this manner, and the extracting of lead was particularly good. The later work of Stansfield and Ingalls also showed the same possibility, and ores of this class are now being treated at Trollhättan.

EXPERIMENTS OF JOHNSON.

One of the most persistent investigators of the possibility of smelting zinc ores in the electric furnace is W. McA. Johnson,[10] who has conducted experiments on a large laboratory scale for several years.

Johnson's furnace has vertical upper carbon electrodes and a conducting hearth of carbon. Ore is fed in through the roof of the smelting chamber. There are several tap holes at different levels. The zinc vapor passes through a flue into an adjacent column of carbon which is heated by the passage of

[10] Metallurgical and Chemical Engineering, **10**, 537 (1912).

the electric current. During the passage of the vapor down through the hot carbon, the carbon dioxide is supposed to be reduced to monoxide. The vapor then passes into a condenser, where most of the zinc is condensed as metal and some as blue powder. The furnace is operated continuously. The ore is preheated by gas to 900° C. before entering the electric furnace.

Except for a few statements, the information printed about the work done by Johnson does not clearly show the extent to which he has carried condensation without the formation of blue powder, but the use of a column of hot carbon to reduce the carbon dioxide in the vapor seems to have assisted in lowering the percentage of blue powder formed.

ELECTRIC SMELTING OF ZINC SULPHIDE ORES WITH IRON AS A DESULPHURIZING AGENT.

The two chief exponents of the use of iron as a desulphurizing agent in the electric smelting of zinc sulphide are the Imbert and Cote-Pierron processes. Imbert tried out the reaction without much success in the combustion furnace. Thomson and FitzGerald later designed an electric furnace for its application. Although the Imbert-Thomson-FitzGerald process has been tried experimentally on apparently a good-sized scale for two years in Upper Silesia, little authoritative information has been given out concerning it. Several types of resistance furnace have been used, all having a resistor of solid carbon.

The Cote-Pierron[11] process is performed in a furnace of the Siemens type, or one having the two vertical electrodes in series, both types being roofed and connected with a column of electrically-heated carbon, through which the vapor passes before going to the condensing chamber.

The same trouble has been experienced in the use of iron as in the carbon reduction method, namely, in condensation a large proportion of blue powder being formed. It seems that iron and iron sulphide are volatilized and other vapors given off which dilute the zinc vapor and cause similar trouble in the condensation of the zinc as in the reduction of zinc oxide with carbon. Even with the column of carbon of the Cote-Pierron process this trouble

[11] Metallurgical and Chemical Engineering, **7**, 468 (1909).

continued. The Cote-Pierron process does not appear to have developed into the commercial success expected of it.

While it is claimed that, by the use of iron as a desulphurizing agent in the direct treatment of sulphide ores, roasting is avoided, it is probable that any saving in this respect would be more than offset by the cost of iron. For carrying out the reaction about 700 pounds of iron are stated to be necessary per ton of 30 percent zinc ore. At $12 per ton the cost of iron would be about $4 per ton of zinc sulphide ore treated. The cost of roasting a zinc sulphide ore is estimated by Ingalls at from $1.25 to $1.75 per ton. It is doubtful if a regenerative process for recovering the iron from the iron sulphide formed would be very successful, or cheap enough to reduce the cost below that of roasting. The iron sulphide might be sold if a market was near, but it probably would be contaminated by other metals in the ore.

PRESENT STATUS OF ELECTRIC ZINC SMELTING.

Although, as previously stated, more progress has been made to date in the electric smelting of zinc ores than with the electric smelting of any of the non-ferrous metals except aluminium and metals forming ferro-alloys such as silicon, chromium and tungsten, nevertheless the process is still largely in the experimental stage. There is no plant operating on a commercial scale except the Trollhättan works taking from 7,500 to 10,000 kilowatts. The experimental work mentioned is still going on, but as yet none of it has developed into a commercial operation.

From what has been said concerning the work at Trollhättan and the results of others, it is very evident, as previously explained, that the difficulty lies almost entirely in condensation of the zinc vapor to a metal rather than blue powder under the peculiar conditions of the electric furnace. The electric furnace, mechanically or electrically, presents no great difficulties to solve, because all of the troubles formerly experienced have been solved in the construction of large pig iron, steel, carbide and ferro-alloy furnaces. The problem, then, is one of a metallurgical nature, and is caused by the different conditions and greater speed of smelting in the electric furnace than in the combustion retort.

The opponents of the electric smelting of zinc ores, who as a rule appear to have no knowledge of the great development of

the electric furnace itself in the treatment of other ores, often state that, while the retort is the weak part of the old system, so is the electrode the weak point of the electric method. Admitting that the retort is the weak part of the retort method, and judging from large scale electrometallurgical work on other ores, we do not believe that in general the electrode is an exceptionally weak part of electric smelting. The electrode problem, no longer a serious one, has been solved in iron smelting. It is only fair to assume that the designer of an electric zinc furnace should stand upon the accumulated knowledge and take the results of others when the metallurgical problems of electric zinc smelting have been solved. One editorial writer in the *Engineering and Mining Journal* of December 14, 1912, goes so far as to say, in reference to retorts and electrodes, "nobody can say which will be the weaker."

While it is not the purpose of the writers to enter into a discussion of the relative advantages of processes, it may be of interest to present a few comparative facts regarding retort and electrode consumption.

Ingalls[12] states that the average life of a Silesian retort over a period of ten years is 38 days. One charge is smelted per day. An editorial in the *Engineering and Mining Journal* of December 14, 1912, states that the Silesian retort takes 200 pounds of ore per charge. Hence a retort treats 7,600 pounds or 3.8 tons of ore. Ingalls[11] also states that a condenser lasts from 8 to 12 days, so that about four condensers are necessary for the ore smelted by a single retort. He also states that a seasoned retort is valued at 50 cents, and a condenser at 4 cents. Thus the cost of a retort and condensers for smelting 3.8 tons of ore is 66 cents, or 17 cents per ton of ore smelted.

At Trollhättan, Sweden, the electrode consumption has been reduced to 6 pounds (3 kg.) per ton of pig iron produced. Considering that two tons of ore were charged for each ton of pig iron produced, the consumption per ton of ore is 3 pounds. The smelting conditions in electric iron reduction would not tend to be as strongly reducing as in the zinc retort, although the ore in each case would be an oxide. Hence it may be expected that

[12] W. R. Ingalls, The Metalurgy of Zinc, p. 545 and p. 242.

the total consumption of electrodes in a large, properly-designed zinc furnace would not exceed 3 pounds per ton of ore.

There is no longer loss of electrodes because of unconsumed ends, as this loss has been entirely overcome by jointing both amorphous carbon electrodes and graphitized electrodes. This reduces the loss by one-half.

In some experiments by the writers on a mixture of sulphide and oxidized copper ores, with no carbon in the charge, and the charge proportioned so as to contain 15 percent sulphur, the electrode consumption with air blown into the furnace for partial pyritic smelting was only 2 pounds per ton of ore. However, the furnace was designed so that the charge protected the electrode from the air.

Amorphous carbon electrodes can be obtained at from 3 to 4 cents per pound, so that, on a basis of a consumption of 3 pounds per ton of ore, the electrode cost would be from 9 to 12 cents as compared with a retort cost of 17 cents. These figures seem to show that, rather than "nobody can say which will be the weaker," the retort at the present time is the weaker.

If the failure of the electric furnace in smelting zinc ores is not due to the electric furnace or the electrodes, what then is the metallurgical condition which causes condensation difficulties and has retarded the commercial application of the process? The difficulty is, of course, due to a difference in the smelting conditions of the electric furnace as compared with the retort. Both physical and chemical conditions influence the formation of blue powder. Some powder forms under certain conditions of pressure and temperature. As to just what these conditions are has not yet been solved. It is a problem for the physical chemist.

The chemical conditions leading to the formation of blue powder are due largely to the speed of reduction of zinc oxide in the electric furnace and to the presence of volatile impurities in the charge. Boudouard found that the permissible percentage of carbon dioxide in the zinc retort was very low, and that when the limit is exceeded the carbon oxidizes the zinc, causing formation of blue powder. The vapor of the zinc retort is usually below this limit or not much above it, so that the zinc is condensed chiefly as liquid zinc. But in the electric furnace the temperature directly beneath the electrode in either the arc or resistance fur-

nace is so high that the reduction proceeds so rapidly that the large amount of carbon dioxide formed does not have a chance to be reduced to monoxide by the hot coal of the charge before entering the condenser. Hence there is the oxidation of the zinc mentioned.

By the use of electrodes of large cross-section it is probable that this concentrated heating could be reduced so that, rather than a very high temperature at one place, there would be a uniform comparatively low temperature all over the charge. This was found to be the case in the smelting of iron ores both at Héroult, California, and Trollhättan. Or the current could possibly be distributed more evenly by flat electrodes of rectangular cross-section.

The formation of blue powder is also caused by volatilization of silicon, which condenses on the zinc either as silicon or silica. For example, in some experiments at the Bureau of Mines Laboratory the condensed fumes from a ferro-silicon furnace contained 67.69 percent SiO_2, 9.72 percent Al_2O_3, 4.11 percent FeO, 0.20 percent MgO, 1.90 percent CaO, with the rest carbon. This volatilization of silicon occurs in any electric furnace operated with an arc, as in ferro-silicon manufacture, when there is silica in the charge. At the lower temperature of a resistance furnace it is not so likely to occur.

The solution of this problem is very probable, and, while it is difficult, there is no reason why it should not be worked out in time. When this is solved, and it is not necessary to resmelt a large proportion of blue powder as at Trollhättan, where two tons of blue powder are smelted for each ton of ore treated, it is probable that electric zinc smelting will proceed rapidly. The use of iron as a desulphurizing agent does not seem to have advanced as far as reduction of the oxide with carbon, and it is probable that the latter will keep its present supremacy. Some work has been done in an electric furnace operated under pressure or vacuum, but only a beginning has been made in this direction.

COMPLEX SULPHIDE ORES.

The electric furnace has a possible field in Western metallurgy at places where there are complex sulphide ores of lead, zinc, copper, gold and silver. With the solution of the zinc condensa-

tion problem it is not unreasonable to suppose that ores of this class can be electrically smelted so as to get a fair recovery of the metals and at least the precious metal values. Also, owing to the fact that practically as high a temperature as desired may be obtained in the electric furnace, it would be possible to smelt complex ores which have high gold and silver values, but which are too low in base metals, such as lead and zinc, to make their recovery worth the additional expense of concentration.

In the blast furnace 10 percent of zinc in the charge causes sticky slags, and with a much higher percentage the operation of the furnace is very irregular and difficult. These slags could be fused readily in the electric furnace. In such a case, if the ores contained copper and zinc the ore could be charged into an electric blast furnace using matte as a collecting agent for the gold and silver.

GOLD AND SILVER ORES.

In the smelting of gold-silver ores carrying no lead or copper, to form either a lead bullion or a copper matte, but containing iron sulphide, the electric furnace might be used in connection with an air blast for oxidation of the iron sulphide, and an iron matte as a collecting agent for the gold and silver. The value of iron matte as a collecting agent is still largely a matter of personal opinion, but from the pyritic smelting which has been done with iron as a collecting agent, and from some experiments of the writers' using iron matte in the electric furnace, it is evident that a fair recovery can be made in this way. As in the case of electric copper smelting, the electric furnace treatment of these ores is still in the speculative stage.

Recently a plant has been installed at Lluvia de Oro,[13] Chihuahua, Mexico, for the electric melting of zinc cyanide precipitate and the smelting of ore concentrates. The gold-silver bullion resulting is shipped, and the slag resmelted or concentrated. The process appears to be successful in its application.

The writers believe that mines in isolated districts, where fuel is usually high and where the power operating the mine or mill is often hydro-electric power, could in many cases melt their cyanide precipitates more economically in electric furnaces than in combustion furnaces. The construction of a small Siemens

[13] Engineering and Mining Journal, June 15, 1912, p. 1189.

type of furnace is simple and inexpensive. By operating the electric furnace when one of the units of the mill was down for repairs, or on the mine shift when little hoisting or drilling was being done, no additional electrical installation would be necessary. This simply means that power which under ordinary conditions is wasted would thus be utilized.

APPLICATION OF THE ELECTRIC FURNACE TO THE SMELTING OF RARE METAL CONCENTRATES.

By rare metals in this instance is meant such metals as chromium, tungsten, molybdenum and vanadium. Chromium is not rare in the sense of the size of its ore deposits, but its metallurgy is so closely allied with the electric smelting of other more rare ores for the production of ferro-alloys that it may in this instance be considered as a rare metal.

The treatment of these ores is of particular interest to the Western metallurgist, for all of these ores are mined in the Western States, but their treatment has been confined to concentration by ore dressing or chemical methods, or shipment of their concentrates to the ferro-alloy manufacturer or the metal refiners. If these concentrates could be treated locally they would be given a "form value" at once, and thus enable the district in which they are produced to retain money which is now expended for freight charges, and also to retain the profits of the ferro-alloy manufacturer, which, because of the specialty of the product, are rather large.

The smelting of concentrates of tungsten, molybdenum and vanadium ores for the production of either the metals or their ferro-alloys presents problems different in one marked respect from the smelting of copper, lead or zinc ores, namely, the temperature necessary for completion of the reduction reaction is so high, or the melting point of the product is so great, that the operation could not be performed metallurgically or economically in a blast furnace, reverberatory furnace or a crucible. Tungsten is reduced from the trioxide at a temperature of 1500° C., and vanadium from vanadate of iron or calcium at about the same temperature. The melting point of tungsten is 2800° C., molybdenum 2000° C., and vanadium 1750° C. All of these temperatures are high compared with that for the reactions usually

performed in combustion furnaces. Of course, the melting point of the ferro-alloy would not be as high as that of the pure metal, and becomes lower with increased percentage of iron.

The only rival of the electric furnace for high temperature metallurgy is the thermit reaction, which is more expensive. The electric furnace soon after its introduction into the manufacture of ferro-alloys rapidly superseded combustion furnaces for this work in all instances except the manufacture of ferromanganese. It was found to be especially fitted for the smelting of rare metal ores to produce ferro-alloys because of the ease with which high temperatures were maintained.

PRESENT STATUS OF THE MANUFACTURE OF FERRO-ALLOYS IN THE ELECTRIC FURNACE.

The growth of the ferro-alloy industry in Europe has been rapid since 1899, but comparatively slow in the United States. There are about 25 plants engaged in the electric furnace manufacture of ferro-alloys in Europe as compared with two plants making the alloys in the electric furnace in the United States. In addition to these there are three companies using methods other than electric furnace methods in the United States, and one electric furnace ferrosilicon plant in Canada.

There are several reasons why the growth of the ferro-alloy industry has been more slow in America than in Europe. Hydro-electric power is not so cheap here and not so favorably located for the receipt of raw material and sale of product. The water-power sites can not be developed as cheaply as many of the foreign sites, where the cost per kilowatt year varies from $10 to $20, as compared with $20 to $40 in the United States. In Canada power is somewhat cheaper, but is often in inaccessible places. Most of the Norwegian and Swedish ferro-alloy plants are located on tide-water or navigable rivers. French works are within a couple of hundred miles of Marseilles. The use of ferro-alloys in the manufacture of high-class steels did not advance as rapidly in the United States as in Europe. Owing to less favorable natural advantages, electrochemical and electrometallurgical industries in general have not made so rapid progress and growth.

A considerable proportion of the ferro-alloys used in the United States are imported, for, although there is a duty, local manu-

facturers are not able to supply the whole demand. About one-half of the ferromanganese and ferrosilicon used in the United States is imported, as well as a large part of the ferrotungsten. More ferrotitanium and ferrovanadium are manufactured here than abroad. The ferrochrome production just about supplies the domestic demand.

In Europe the industry of ferro-alloy manufacture in the electric furnace is in excellent condition commercially, with the demand for alloys steadily increasing. Because of the race between foreign countries in the building of large navies, there is great demand for ferrochrome. The sale of ferro-alloys in Europe, especially ferrosilicon and ferrochrome, is accomplished by the various plants combining in a syndicate, with each plant receiving a certain portion of the total market demand, according to the plant capacity.

GENERAL CONSTRUCTION OF ELECTRIC FERRO-ALLOY FURNACES.

In general an electric furnace for ferro-alloy manufacture consists of a crucible without a roof, having either a conducting or a non-conducting hearth and an upper vertical carbon electrode or electrodes. Most of the furnaces first built had a conducting hearth, and this practice is still common in furnaces up to 750 kilowatts' capacity, but for larger sizes experience has shown the non-conducting hearth type to be preferable. One reason for this is because there is considerable loss of electrical energy by dissipation through the lining, and hence there is a tendency to have a lower power factor. In working a small furnace intermittently, *i. e.*, discharging completely before charging again, the conducting hearth furnace is much easier to operate and start cold. When a large furnace is to be operated for several months continuously on one alloy, this advantage of the conducting hearth is of no importance. A factor which has been little considered in the construction of ferro-alloy furnaces is the power factor, as they are generally completely enclosed in sheet steel, when bands would do as well, with much less induction resulting in the circuit.

The ferrosilicon furnaces are lined with carbon, while the furnaces to be used in producing other alloys are generally lined with a basic lining of magnesite or dolomite brick or a mixture of tar

with either material. When low carbon ferrochome is being made the lining is sometimes chromite. Generally a furnace is designed so that the electrode or electrodes are at a sufficient distance from the sides to permit a chilling of the material charged upon the walls. This has been found to give the most satisfactory lining. Under such conditions furnaces have been operated for two or three years without being relined. The average furnace operated continuously runs about two years before being shut down for repairs. A furnace operated intermittently must, or course, be relined more often.

Owing to the greater part of the reduction of oxides in ferro-alloy manufacture being done at a high temperature by solid carbon, most of the furnaces are run with an open top, into which the charge is placed around the electrode. Under these circumstances there is a considerable waste of heat which might be used in preheating the charge. In these furnaces the electrode usually dips about 12 to 15 inches beneath the top of the furnace, which is kept filled with cold material so as not to expose the molten mass beneath. There is burning of carbon monoxide around the electrode, and an escape of a large volume of gas through the top of the furnace house. It is stated that one of the Alby type of carbide furnaces is operating with a closed top at Kopporaaen, Norway. The Helfenstein furnace does away with the open top also, as it is air tight, and in addition has feeding shafts where the charge is preheated by the waste gases. In the manufacture of the higher-priced alloys, such as ferrotungsten, it is impossible to use any furnace but one with an open top because of the intermittent nature of the operation, but in making ferrosilicon and ferrochrome it seems advisable to preheat the charge as much as possible and prevent as much oxidation of the electrodes as is possible in this manner.

A large number of the first ferro-alloy furnaces were supplied with direct current when the furnace house was built adjoining the power house, so that there was only about 20 feet between the furnace and the generator. It was soon found that the electrolytic action in a direct-current furnace resulted in deposition of potassium, sodium, calcium, aluminum with the alloy so that as pure a product could not be obtained with direct as with alternating current. When alternating current was first used,

single-phase current was the more common in general application, so that the arrangement remained the same as previously, the generator delivering directly to the furnace at the desired voltage without any transformation.

Later, when three-phase current became prevalent, furnaces designed to take single-phase current were connected in groups to three-phase systems by several methods. Then the single-phase furnaces were replaced for large scale work by the present-day three-phase furnace.

COST OF POWER.

Naturally, all other conditions being favorable, the use of the electric furnace in the smelting of ore and the production of metals and ferro-alloys will depend largely upon the cost of electric power.

Hydro-electric Power.

Ordinarily we think of electricity as being most easily developed by water-power. In a way this is true, but as to whether power can be as easily and cheaply produced in this manner as by steam or gas will, of course, depend upon the initial cost of installation, which in turn depends, among other things, upon:

1. Mean average flow of water for twelve months.
2. Whether expensive reservoirs have to be constructed.
3. Whether these reservoirs are situated a long distance from the site of the power plant, thus necessitating the construction of long ditches, flumes, tunnels or pipe lines.
4. Whether expensive dams have to be constructed.

In all the water-powers which are available, either one or all of the above mentioned prerequisites have to be met and dealt with, and the cost of installation may be as low as $48 per kilowatt or as high as $400. As shown in Table II, the cost of hydro-electric power to the consumer may vary from $5 to $60 per kilowatt year, depending largely on local conditions.

Power Produced by Gas Engines.

Of late years a great deal has been said and written as regards the production of cheap electric power by gas-engine-driven generators. It is quite true that power can be produced cheaply in this manner when waste gases from a blast furnace are utilized. On the other hand, if coal is burned in gas producers to produce

the necessary gas, and even if the coal used is of an inferior quality, and a by-product plant is operated in connection with the gas-producer plant, the cost of electric power made in this way is quite likely to be more than is generally supposed it can be produced for where gas engines are used for driving the generators.

As to what the cost of power produced by gas engines would be, a reliable engineering concern has furnished data which show that, with coal at $3.00 per ton, which it is assumed will contain 13,000 b. t. u. per pound, electric power can be produced at about the following cost when using gas-engine-driven generators at a load factor of 75 percent:

Gas-engine-driven Generators.

Size of Plant, Kilowatts	Cost of Power in Cents per Kilowatt Hour, Coal at $3.00
2,000	0.745
4,000	0.687
6,000	0.649
8,000	0.637
10,000	0.633

These figures include operating costs and fixed charges.

Power Produced by Steam Turbines.

The same engineering concern has also supplied the writers with data in regard to the cost of electric power produced by steam-turbine-driven generators. Based upon this data, the cost of electric power is shown in the following table, with coal at $3.00 per ton. These costs include operating costs and fixed charges, and, as before, it is assumed that the coal has a heating value of 13,000 b. t. u. per pound and that the load factor is 75 percent.

Steam-turbine-driven Generators.

Size of Plant, Kilowatts	Cost of Power in Cents per Kilowatt Hour, Coal at $3.00
2,000	0.688
4,000	0.597
6,000	0.532
8,000	0.526
10,000	0.514

In the following table are given the approximate costs of power per kilowatt year at various electric furnaces and electrochemical plants in this and other countries:

TABLE II.

Cost of Electric Power for Electric Furnace and Electrochemical Purposes with a High-load factor.

Location	Power Company	Nature of Power	Cost per Kilowatt Year	Product at Present Time
Braintree, England	Lakes & Elliott Steel Foundry	Gas Engine	$110	Steel
Sheffield, England	Municipal System	Steam	87	Steel
LaPraz, Savoie, France	Electrometallurgical Co. of France	Hydro-electric	16	Aluminum, steel, carbide
Livet, Isere, France	Keller, Leleux and Co.	Hydro-electric	13	Ferro-alloy
Ugine, Savoie, France	Girod Electrometallurgical Co.	Hydro-electric	18	Ferro-alloy, steel
Bonn, Germany	Municpal System	Steam	92	Steel
Remsheid, Germany	Lindenber Steel Co.	Steam	87	Steel
Volklingen, Germany	Rochling Iron & Steel Co.	Steam, Gas	83	Steel
Meraker, Norway	Meraker Electric Smelting Co.	Hydro-electric	8	Ferro-alloys, carbide
Notodden, Norway	Norwegian Saltpeter Co.	Hydro-electric	7	Nitrates
Rjukan, Norway	Norwegian Saltpeter Co.	Hydro-electric	5	Nitrates
Tyssedal, Hardanger, Norway	Tyssedal Power Co.	Hydro-electric	10	Carbide, cyanamid
Trollhattan, Sweden	Royal Power Plant	Hydro-electric	$9 to 12	Iron, zinc, ferro-alloy, paper
South Vernon, Vermont	Connecticut River Power Co.	Water and Steam	53	General
Shelburne Falls, Mass.	Connecticut River Power Co.	Water (Secondary)	29	General
Turners Falls, Mass.	Turners Falls Power Co.	Water and Steam	60	General
Turners Falls, Mass.	Turners Falls Power Co.	Water (Secondary)	26	General
Rumford Falls, Maine	Rumford Falls Power Co.	Hydro-electric	26	Paper
Niagara Falls, U. S. A.		Hydro-electric	26	Electrochemical
Niagara Falls, Canada		Hydro-electric	20	Electrochemical
South Carolina	Southern Power Co.	Hydro-electric	25	Nitrates
South Chicago, Ill.	Illinois Steel Co.	Steam, gas	43	Steel
South Carolina	Southern Aluminum Co.	Hydro-electric	25	Aluminum
Snake River, Idaho	Southern Idaho Power Co.	Hydro-electric	12	General
Heroult, Cal.	Northern California Power Co.	Hydro-electric	16	Pig Iron

CONCLUSION.

In general, then, we may say that, aside from the smelting of iron ores, the manufacture of aluminum and the production of ferro-alloys, the electric smelting of ores is still in the experimental stage. It will probably never replace existing processes where the conditions are favorable to the latter, except in so far as it may permit of the development of a process which may be more efficient than an existing process. For example, in the retort process of zinc smelting, a furnace temperature of from 1400° to 1500° C. is required. This is the temperature on the outside of the retorts, whereas the actual temperature inside the retorts is from 100° to 200° less than this. Therefore, in this respect an electric furnace would offer the advantage that the full temperature of the furnace could be had just where it is needed, that is, where the work is to be done. For that reason, even though the electric current may cost more than coal for an equivalent heat value, the electric furnace may nevertheless be found more efficient and hence in the long run more economical than the combustion furnace, especially if the current can be used in the manner just indicated. However, as we have repeatedly stated, we do not believe that it is necessary to consider the electric furnace as a competitor of the combustion furnace, but as affording the metallurgist a furnace which will enable him to treat certain ores and to work out certain processes in a more advantageous manner than it is possible to do in the combustion furnace, or to treat ores in certain districts where owing to local conditions it would not be commercially feasible to treat such ores.

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DISCUSSION.

F. A. J. FitzGerald (*Communicated*): This paper may be fairly considered as official or at least semi-official, as it is "published by permission of the Director of the U. S. Bureau of Mines." Presumably the object of the Bureau of Mines is the carrying on of work more or less related to mining, for the

benefit of the people whose pockets supply the very large appropriation necessary for the maintenance of the Bureau. This being so, no more valuable public service can be performed by the American Electrochemical Society than the careful studying of the work of the Bureau in so far as it has any bearing on electrochemical subjects, with the object of offering criticisms which will improve the efficiency of the Bureau and enable it to render service which will be of value to its employers.

As regards the present offering of the Bureau of Mines it appears that the title of the paper is by no means satisfactory, as it does not represent what the paper really is, *viz.*, an abstract or review of certain work done on the smelting of iron and zinc ores in the electric furnace, reference to the possibility of smelting copper, lead, gold and silver ores, and a few remarks on the ferro-alloy industry. Surely a paper corresponding to the published title should contain more than this one does about the conditions of Western metallurgy and their relation to the possible application of the electric furnace. If the metallurgical section of the U. S. Bureau of Mines proposes to make a study of the applications of the electric furnace it is of course necessary that it should be thoroughly well informed as to all valuable work done by others in this relation, but that it should spend its time making abstracts or reviews does not appear to be economical. Abstracts and reviews when desirable can be obtained in a cheaper way.

An interesting reference is made to "a preliminary investigation conducted by the metallurgical section of the U. S. Bureau of Mines on the possibility of using crude oil as a reducing agent." It would be very interesting to get some information on the economic value of this proposition. It is possible that a method might be worked out for the use of crude oil as a reducing agent and it might be cheap; but is it truly economical? An enormous number of the employers of the Bureau are painfully conscious of the demand for crude oil and will they be benefited by the Bureau's discoveries of new uses for this material? At least they would like to be shown that it conforms with the great principles of conservation. In this relation it would also be interesting to have more information on the relative value of coke and charcoal

for reduction purposes, because the reasons given for the superiority of charcoal are by no means convincing. Is it not possible that the furnace design has something to do with the results obtained?

There are many other references in this paper which call for comment and discussion, but its apparent object is the consideration of problems connected with Western metallurgy, a subject with which many of our members are by no means familiar. For this reason I do not think it desirable to take up further time which should be devoted to the views of those better acquainted with the subject, as these will not only be of interest to our members, but will doubtless greatly assist the Bureau of Mines in the important work which it has undertaken.

D. A. Lyon: I am very glad that Mr. FitzGerald has contributed this discussion. In the first place I notice he states that no more valuable public service can be performed by the American Electrochemical Society than in studying and criticising the work of the Bureau on electrochemical subjects. That is quite true. I cannot speak officially as far as the Bureau is concerned, but I am sure the Director of the Bureau will be more than glad to receive friendly and instructive criticism of our work, and with that end in view I hope the American Electrochemical Society will appoint a committee to confer with the Director in regard to what the Bureau can do in order to help electrochemical and metallurgical work along in this country, and that whatever we may do may be along a line that will be in accordance with the views of this Society, and which will be for the best interests of the industry as a whole.

In regard to the paper under discussion; it is, in a sense, an abstract of that which has appeared in print up to the present time in regard to the various applications of the electric furnace in metallurgical work, and yet, at the same time, had Mr. FitzGerald read the paper more carefully he would have found that the authors had incorporated their own views in the paper as well. However, for the sake of argument we will grant that the paper is simply an abstract. The view that I hold in regard to such work is that there is a certain amount of information with which the public needs to be acquainted. In other words, as

Dr. Holmes, the director, has stated it, the function of the Bureau is to ascertain facts, not necessarily at first hand, but to ascertain them and then disseminate them to the public—and by the public is meant the ordinary layman and shop man as well as the technical man. I was impressed with this fact at our last meeting. I believe that about 99 percent of our papers might just as well go into the waste basket as far as the general public is concerned. It seems to me that the Bureau must take into consideration the fact that the layman as well as the technical man needs information, and that one of the best services which the Bureau can perform is to keep the public informed as to the status of any particular line of work in which they are interested, and especially as regards new subjects, such as electric furnace work. Of course work of this kind will not be of any particular value to men who are experts in that particular subject, as is the case with Mr. FitzGerald in electric furnace work, but it is hoped and expected that it will be of assistance to those who, for one reason or another, are not familiar with the subject, and who have not had time to follow it closely. That is why we offered this paper at this meeting, for we assumed, and we believe we are right, that there are members of this Society who have had no reason for following closely the subject of the use of the electric furnace in metallurgical work, but who are nevertheless interested in the subject. We also thought that it might be well for the Society to contribute something to the literature on this subject which would be understood by anyone who was enough interested in the same to read the paper. Therefore, for reasons which I have tried to point out, it is quite likely that the Bureau will do a great deal more of the kind of work which Mr. FitzGerald calls abstracting, and will publish the same from time to time as they may think best.

As to the other points taken up by Mr. FitzGerald, he states that an interesting reference is made in the paper to a preliminary investigation which has been made by the Bureau on the possibility of using crude oil as a reducing agent, and that he thinks it would be very useful to get some information on the economic value of this proposition. In reply to this statement. I would say that in California there is a large amount of iron ore waiting to

be treated; the use of coking coals is out of the question. I have recently looked into the matter; a large amount of residuum oil is produced by the refiners for which, I am informed, there is no demand, and as they cannot use it for anything else it is used for making roads. When I was out there they came to me and said, "Can't you find some way of using this residuum oil?" We are trying to do so, and one way we think we can use it is for the smelting of iron ore, and so help to create an iron and steel industry on the Pacific coast.

I have already mentioned the relative value of coke and charcoal as a reducing agent in the electric furnace, that we need more information along this line, and that we expect to get it. It is to be remembered that it has been only two or three months since the plant at Hardanger, in Norway, shut down because they found they could not use coke as a reducing agent. In California they have found out the same thing; that is, that they cannot use coke alone, due to the high conductivity of the same.

It is also to be remembered that the iron furnace is a resistance furnace, that the heat is created by the resistance which the charge offers to the current, and that as coke is a better conductor than charcoal, less resistance is offered by the charge when coke is used as a reducing agent, and hence the current consumption per ton of metal produced is greatly increased. However, as before stated, we still need more information on this subject and hope very soon, as a result of some experimental work which we are contemplating, to secure the same.

As to the paper being weak in regard to Western metallurgy, I admit that this may be true; I am a Western man, and am more or less familiar with the conditions of the West, and it never occurred to me that the other fellow would not be equally familiar.

J. W. Richards: I note in Dr. Lyon's paper an absence of reference to one possible application of the electric furnace to Western metallurgy, and that is the possibility of the electric furnace producing the steel castings necessary in the mining industries. I believe that the steel castings used are brought from the East at a large expense and at a great expenditure of time in getting them.

As an illustration, I will cite a little plant twelve miles from

Bethlehem, Pa., that is operating on a city power supply, using the power between 12 o'clock at night and 4 o'clock in the afternoon, paying 1 cent per kilowatt-hour, melting cold charges of all sorts of scrap iron and steel and making steel castings for the trade in competition with open-hearth castings. If that is possible in the East right among the manufacturers of open-hearth castings, how much more profitable would it be out West with your cheap electric power, taking into consideration the great distance you are from the ordinary producers of steel castings. I believe it would be quite feasible and possible to start that industry and to make for the West all the steel castings you need in your mining industries.

Incidentally I have been told that the railways are the chief objectors to the starting of these local industries. That might have been so in the past, when they possibly had the short-sighted idea that to foster local industries would reduce the amount of freight from the East; but if that proposition is looked at in a broader way, the fostering of these local industries will really in a short time very largely increase the business of the railroads.

In regard to some statements of Dr. Lyon as to the present status of electric zinc smelting, the general belief of those otherwise well informed on the metallurgy of zinc in this country is that it is in an experimental stage. Dr. Lyon has been at Trollhättan and has spoken of the works running on from 3,000 to 10,000 kw.; there is nothing experimental about that, and they have enlarged their plant recently. There is also a plant at Sarpsborg, in Norway, which is operating successfully. When in Europe in July of this year I heard, to my surprise, that there was also another plant, not only projected but installed and in operation at Helsingfors, in Finland. The present plant is 2,500 horse-power, and it is being enlarged to 6,000 horse-power. The reason the builders of the plant are so secretive is that they have found it so profitable that they want to keep other people out. I therefore think that the actual state of the matter is that the electric furnace zinc industry in Europe is being very profitably worked. Our experimenters in this country are considerably behind them.

I wish to dissent from a statement which Dr. Lyon ascribed

to Mr. Johnson that the electric smelting of zinc had gotten so far that it was now more an engineering problem than a metallurgical one. No greater mistake can be made by anyone working in electrochemical lines than to think that their problem is merely an engineering problem. The steel industry is just recovering from that misunderstanding. I have been told many times that the working of an open-hearth steel furnace is only an engineering problem, that they have no metallurgical questions to solve there. There can be no greater mistake; the chemist or the metallurgist is using his chemistry all the time, and when he thinks he is down to merely an engineering problem he is making the mistake of his life.

I would like to ask Dr. Lyon if the treatment of two tons of blue powder per ton of ore smelted, at Trollhättan, is not a mistake?

D. A. Lyon: No, I think that is how it is given.

J. W. Richards: That would mean that they treated the same powder over several times?

D. A. Lyon: Yes.

C. A. Hansen: I have been giving all of my time during the past eight years to electric furnace work with the General Electric Company, and I have had in mind for a long time looking up some of the better possibilities of development for that class of work in the Western States. The main reason for my coming here was to hear what Johnson and Peterson had to say about zinc, but now I am asked to read their papers.

I happen to have had quite a little to do with the particular electric steel plant to which Dr. Richards refers. They do not, at that plant, make castings from any old scrap that comes along. They could undoubtedly do so, but not at a final cost that would compare favorably with their present practice of using boiler plate punchings, and similar good quality scrap. They do not, at that plant, try to compete with the open-hearth furnace. They are making castings of an average weight of something like ten pounds, castings that in general the open hearth cannot take over in job lots.

It may be of interest here to set down some figures for molten steel costs within 200 miles of New York City. These figures

apply to casting steel and are taken directly from actual cost records. The labor and material conditions are quite comparable with one another. Cold metal charge was used in each case. Power was charged at 0.8 cent per kw. hour to the electric furnace.

	Acid OH Furnace	Tropenas Converter	Crucible	Electric Arc Furnace
Size of Furnace	25 tons	2½ tons	4 x 12 pots	5 tons
Working day—Foundry	24 hrs.	24 hrs.	24 hrs.	9 hrs.
Working day—Furnace . .	24 hrs.	24 hrs.	24 hrs.	12-13 hrs.
Average daily output of Molten Metal	100 tons	20 tons	10 tons	10 tons
Cost per ton of Metal in Ladle (2000 lbs). Metal and Alloys	$15.44	$20.65	$17.00	$11.29
Total cost	20.65	26.18	58.10	24.28
Percent of Molten Metal in good Castings	61	54	64	59
Percent Carbon in Steel	0.25-0.30	0.30-0.40	0.35-0.50	0.15-0.25
Average Weight of Castings . . .	260	80	15	15

For the electric furnace, power amounts to 20.7 percent of the cost of molten metal and 6.8 percent of the total cost of castings.

So far as limitations for the various processes go, the more important are:

Open Hearth.—Large furnace and large output are necessary to reproduce the costs shown. Continuous operation essential to maintenance of furnace plant. Difficulty of distributing metal in small capacity molds from a large capacity ladle. In one case that I know of, a five-ton electric furnace, taking all castings below 50 lbs., increased the average weight of the castings of a 25-ton open-hearth furnace from 80 to 260 lbs. and reduced costs of both light and heavy castings as a result.

Tropenas Converter.—The plant referred to was discontinued because of irregularities of output, as many similar plants have been discontinued.

Crucible Furnace.—$20 to $22 per ton of metal melted, as crucible cost alone, is not uncommon where the attempt is made to make castings under 0.30 percent carbon, nor is 300 gallons oil per ton melted uncommon in crucible practice.

Electric Furnace.—Practically no limitation so far as steel composition goes. The cost limitation is not so serious as it is usually

supposed to be. It will not compete in cost with the open hearth on relatively heavy work except under exceptionally favorable conditions, but it will compete with the open hearth on light castings or thin sections generally.

It may very well be that there are places in the West, perhaps Butte, Montana, or Great Falls for instance, where there are heavy scrap accumulations so far removed from market that the price is low, and where power is reasonably cheap—say 1 cent per kw. hour—and where a market exists for castings. In such places it will pay to look into the electric steel process.

One of my duties, at times a major one, is to answer inquiries regarding the commercial feasibility of this or that electric furnace development. One class of applicants for information, relatively a large one so far as the West is concerned, is the power companies which have a lot of excess power to dispose of. Most of them want some outlet for power to tide them over a period of years while a more profitable load is being developed. Many of them want to know whether or not a steel plant would pay. The main difficulty is that a 1,000 kw. load in a steel castings plant means something like a quarter million dollar foundry, *i. e.*, many of the possible outlets for power require such excessive capital expenditures that mere power cost is not at all the determining factor. Such propositions should interest this class of power companies only as regards their permanent load development. Ferro silicon, calcium carbide, etc., are products in which power, freight and market are the determining features. The cost of a plant using several thousands of kilowatts is only nominal but in the West you have no market short of $15 to $20 freight rate on a $60 to $80 product. If a few points quite as elementary as the above were even considered before an inquiry were sent in, my work in this direction could very easily be made a labor of love instead of an abomination.

Another class of inquiries deals with the electric smelting of galena in some remote locality. Galena is very easily concentrated to about 80 percent lead, and the concentrate is very easily smelted by well known means. Can it possibly be so serious a matter to pack out 100 pounds of galena instead of 80 pounds of lead, that the commercial success of the proposition hinges on the development of an electrochemical process? I do not believe it.

The third large class of inquiries deals with electric smelting of zinc ores. I believe firmly that electric zinc smelting has a large field, but here again power is not the determining factor. I am at present on my way to California to compare the possibilities of an electric furnace zinc process developed in the East with those of an electrolytic zinc process developed in California. We think we can smelt this particular 4 percent copper, 25 percent zinc, $1 gold and silver ore with power at 0.4 cent per kw. hour, at a profit of some $7 or $8 per ton—using the electric furnace. In this case, as I remember the figures, power amounts to $4.00 to $4.50 out of a total smelting cost of about $20.00.

It does not follow, however, that anything so marvelous attaches to electricity as applied to zinc that the electrochemical processes are going to make bonanzas of every 2 percent carbonate ore, etc., carrying no other appreciable values.

Finally, I want to help where I can, and the General Electric Company wants to help, but I do wish that people who ask for our advice would realize that they are in far better position to size up their problems on the ground than are we at a distance of a few thousand miles. A little reflection could then boil down the inquiry to something specific to which one could make an intelligent reply.

G. H. Clevenger: It is a well-known fact to those who are familiar with conditions at Butte that all the scrap iron and steel for miles around is kept cleaned up by the demand for it for the precipitation of mine water. This applies to everything from rails to tin cans. On the Pacific coast there is not, as many people suppose, a great supply of steel scrap which can be readily obtained. This doubtless comes about from the fact that there has been in operation in South San Francisco the open-hearth steel plant of the Pacific Steel Company. This, together with a number of other installations for the making of steel castings from scrap, has pretty generally cleaned up the scrap in this locality. In fact, the situation regarding the scrap market is such that the Pacific Coast Company has been looking to other sources for at least a portion of their raw material. In this connection I believe that they are carrying on experiments upon reduction of iron ore by oil. In fact, I know of no locality in the West where large amounts of steel scrap would be available.

J. A. MATHEWS: I have heard the tariff described as a local issue. From the previous discussion, I would imagine that the electric furnace might be so considered. Speaking of the electric furnace in the iron and steel trade, Dr. Waldberg, of Sweden, told me recently that he considered the electric furnace in Sweden would have very little application in the melting of steel and its primary use would be for the smelting of ore; yet, in this country, it is my opinion that the electric furnace will be used chiefly for steel manufacture. In a few localities it may be of commercial value in the reduction of ore, but I do not think as large an industry will develop in that line as in the manufacture of steel. Where used in the manufacture of steel, there are three fields in which it is likely to be successful. First, in the manufacture of the highest grades of steel to compete with crucible steel both in quality and price, and also for structural purposes such as automobile parts where open hearth or Bessemer steel is not entirely satisfactory. The second promising application would be the process tried near Chicago by which, without very great expense, Bessemer metal can be refined and rendered more dependable for rails. The third application, and possibly one of the largest, will be in steel castings, which Dr. Richards has referred to. I have had no experience in the manufacture of steel castings, but it seems to me that the results cited by Mr. Hansen favor the use of the electric furnace. Large open-hearth furnaces can be used very economically for large castings, but for small castings the difficulties attending its use are great and can be best overcome by the use of the electric furnace, which can be used in melting cold scrap or for refining liquid metal. You can handle two or three ton charges as they come along, for light castings, and there should be no question about it; from the results that I have seen I see no reason why they should not be almost as cheap as open-hearth castings and vastly better in quality.

We have been operating the electric furnace for seven years for making very high grade steels and, at the present time, we have no reason to think that the electric furnace cannot compete in quality with any grade of steel made by the crucible process.

E. L. CROSBY: I am connected with the sales department of the Detroit Edison Company, and the statements of Dr. Lyon and Mr. Hansen are particularly interesting to me.

The recent advance of crude oil has brought to the Edison Company a vast amount of inquiries from the automobile industry as to some methods of superseding the combustion furnace. At that time we received inquiries from four concerns in regard to replacing the open-hearth by electric furnaces; the matter was turned over to me and I communicated with every central station in the country; I could not get any information from them, except that some of them had furnaces for their laboratory work. The information I have gained from Dr. Lyon is invaluable to us, and it was only upon my becoming a member of this Society that I received any information that would give us anything definite in regard to the cost of electric steel.

The trouble we now have is that nobody is selling an electric furnace. We have three people in Detroit who would make an investment if they could purchase a furnace that would operate, and the Detroit Edison Company has gone to the trouble of establishing a rate of something like $50 a kw. year for such purposes, and we are anxious to obtain such information as we have been getting here.

I think if more publicity were given to the Transactions of this Society, among central station men, and the Society got in touch with them more thoroughly, it would be of vast advantage.

L. B. Skinner: We have had the question of "complex sulphides" at every meeting of every technical society in Denver for the last four or five years. We have heard all sorts of press stories about the enormous quantities of complex ores. I happen to be connected with a concern in the market for these. This company has spent a considerable amount of time trying to find the quantities which some of you may have been misled into thinking exist.

The complex ores in the State of Colorado are mostly amenable to mechanical separation at a very low cost. If you take a complex ore, one-third of it having a zinc smelting content, you have to figure about what the other various parts separated or the other smelting constituents would demand in the way of cost of treatment. The copper and lead smelting concerns have a very low smelting cost, and I would imagine that a $12 per ton smelting cost for zinc ore would be a fair average, this including the roasting as well as smelting of the ore.

At the present time the smelting of zinc ore demands a high grade concentrate, which can now be made mechanically out of these ores, and, if you will average up the cost of treating the mineral contents of these complex ores, you will find you can never approximate it by using the figures quoted in electric furnace investigations. A cost allowance of $26 per ton seems to me prohibitive, and I therefore take exception to what Mr. Hansen has said about the possible margin in treating our Western complex ores.

With respect to steel castings in Denver, I know that that has been considered within the last three or four years. There is not a large market for steel castings, so far as my information goes, to keep a furnace operating regularly. In the second place, Dr. Richards spoke about securing power at one cent per kw. hour in some city. There is no power sold in Denver at one cent per kw. hour; the whole situation with respect to electrothermal and electrochemical work in the West, or as far as Colorado is concerned, is dependent on securing what you people in the East consider a fair price for the power. There is a lower price than one cent per kw. hour in the State of Colorado in the Telluride district where there is some competition, but in other parts where the controlling factors are pretty well united there is no such price as one cent. In Butte the prices are lower; but still it does not approximate anything that I have heard of in the electrochemical industries in the East. Along with this power cost, the much higher cost of plant in the West (since all the apparatus is either made in Schenectady, East Pittsburgh or some other Eastern city) and much higher priced labor for installation, makes us lose our enthusiasm after the first effort.

I know of one concern in the zinc smelting business that is prepared to put in a quarter of a million dollars at any time that the electric smelting of zinc or complex ores is shown to be a success.

There is no great quantity of complex sulphides, but there is a great quantity of what we call low grade carbonates having no metal value other than zinc. It seems that in the formation of these carbonates that the constituents have united so that any mechanical separation is impossible. If this electric zinc smelting

can be done as cheaply as the figures I have noted, these carbonates would be amenable to such electric smelting treatment.

The zinc smelters are now accepting carbonate ore that will run 30 percent zinc at prices which will net $10 at the mines now, but the miners want us to improve these conditions. In handling low grade complex sulphides, our problem has been at all times to try to meet the miner half way, and we, with other people engaged in the ore business in the West, have been looking into this electric furnace proposition in all seriousness. When you analyze figures and you get the data that you cannot hope to get inside of several months' time of hard work canvassing the situation, I venture to say that a great deal of enthusiasm will be lost after that several months' work. The problem is very interesting to me, but I cannot agree with some of the statements that have been made that we understand a great deal about it. I, for one, question the chemistry. No one that I know of knows very much about the chemistry or even the physics of possible zinc vapor combinations. In other words, at the temperatures ordinarily used we have no actual analytical work that has been done on the possible combinations of the volatile zinc, but, since we do not, we are in possibly the same shape as suggested by Mr. Lidbury's criticisms of the lack of necessary data in another paper. This has been the trouble with electric furnace work. As a consequence we have the formation of "blue powder" instead of "metal," and there is a great deal that must be studied with respect to that formation in electric furnace investigations. What is the result of carrying on distillation with a residual mass of material, apparently inert, but which may not be inert? As a result of some few experiments which I have conducted, I have found that the composition of this mass has a very important bearing on the condensation of the metal itself.

Now, to come back to the cheap processes of mechanical separation. With the advantages offered in the way of electrostatic and magnetic separations that have been perfected, and working with a cost for each part or constituent which is within reason, and with a very low cost of smelting the galena and copper and iron products by themselves, it will be difficult to equal the present cost per ton of crude ore with any electric smelting furnace handling ore as mined.

I think that the conclusion of those here who want information will be that there will not be any great change in the situation, and that the electric furnace, as a factor in the treatment of complex ores, has not yet been subjected to enough vigorous analytical work.

C. A. HANSEN (*Communicated*): I stated that I was in a state of ignorance of Western conditions except in so far as I had gleaned them from the correspondence, but that I hoped to remedy this to some extent during the next two months. I am grateful for Dr. Clevenger's remarks, therefore, as being the sort of information I am seeking to collect.

With reference to Mr. Skinner's remarks, I had the pleasure of rather an extended talk with Mr. McGregor, who is connected with electrostatic separation work. I asked Mr. McGregor why it was that his company or one of its licensees, who owned a Wisconsin blende deposit, erected an electrostatic plant for customs ore treatment, but continued to roast and magnetically separate its own ores, and why the electrostatic plant, for which certain claims had been made in print, was not rebuilt after it burned down some two years ago. Mr. McGregor explained this to my satisfaction, and offered to make the same explanation publicly before our meeting. He also volunteered the information that his process was incapable, according to actual test, of commercially treating the California complex ore, with which my remarks were more particularly concerned. I do not think that the publication of the fact that electrostatic separation was not a commercial success on Wisconsin and on these particular California ores is at all a reflection upon the general process. I do not think that the electrothermic process is necessarily generally applicable to all zinc ores, much less to all ores of any description whatever.

We all know, or should at least know, that no single process has ever proved a panacea for all metallurgical ills, although very many processes have been and are of inestimable value in commercial treatment of a particular type of ores.

I firmly believe that one gains more in the estimation of thinking, practical men when one frankly states that his process has fallen down on this, that or the other proposition, and gives some reasons for his failure, than by bluffing it out in generalizations.

I have known able men to turn down an otherwise good process because of extravagant, ignorant and impossible claims for the process.

I am personally of the opinion, after seven or eight years' constant consideration of electric furnace matters, that the development of electric furnace work has been held back several years by many superficial, silly published remarks by men who should know better, some of them in our own Society, concerning the applicability of electric furnaces, or even of electrochemistry generally, to some of our everyday problems.

It has also been held back to some extent by the fact that conclusions are often drawn very positively from work on a ludicrously small scale, by incompetent or at least inexperienced men, at a total cost of, say, $50 or $100 for the whole research, whereas positive conclusions could not and would not have been drawn had $1,000 or $2,000 been wisely expended in the same research. One should hesitate, to say the least, before going on record as regards important conclusions. One should consider whether, after all, there are not some nine chances in ten that a diametrically opposite conclusion could as well be drawn from the data at hand.

With reference to some of the remarks regarding the chemistry of zinc vapor, it may be true that we can learn much by studying it.

On the other hand, the physical constants of zinc vapor may be had in the works of Barus and others.

For the rest, if such oxidizing agents as CO_2, H_2O, SO_2, which may easily be present together with zinc vapor, are rigorously avoided, one will not condense "blue powder," as it is ordinarily termed. If the condenser temperature is too low, it is perfectly possible to condense a zinc "snow" which will flow together when heated to above 450° C.

If zinc vapor is present in the furnace gas to the extent of some 40 percent of the total gas volume, then a 70 percent direct spelter recovery is perfectly possible.

All of this is merely common chemical sense, not theoretically merely, but practically so.

If our friends in Sweden or Norway are getting two tons of blue powder to treat for each ton of ore smelted — I do not

believe this, by the way—they should seek a competent chemist at their early convenience.

L. B. SKINNER: With respect to Mr. Hansen's contribution, my understanding was, in making the remarks I made about the limited tonnage, that he referred to this immediate section of the country. I understood Mr. Hansen to state that he was in Denver at this meeting and was going to remain here to look into the possibilities of zinc smelting in this locality. With respect to the Huff process, Mr. McGregor has already said that the Huff process does not cover the entire field of possible mechanical separation.

LOUIS SIMPSON (*Communicated*): On page 145 of the paper by Messrs. Lyon and Keeney, entitled "Possible Applications of the Electric Furnace," certain statements are given under the authority of "a reliable engineering concern": the name, however, of the concern is not given.

These statements purport to be the cost of producing electric power from power developed in gas engines using producer gas, and in steam turbines.

It is necessary to point out that, since no inconsiderable portion of the cost of power production consists of interest and depreciation charges upon the initial cost of the power installation, and as these charges vary, often within very wide limits, in different countries, according to the cost of the several installations in these countries, and, further, that, as the American Electrochemical Society counts among its members citizens of many countries, the publication of statements of this description, based upon the authority of persons unknown, even if they be connected "with the reliable engineering concern," is to be deprecated.

It is admitted by everyone that figures may be made to prove anything, when manipulated by clever men, anxious to prove something in which they have an interest. No calculations are more susceptible to manipulation than are the calculations of the cost of power; hence it is important that data should in all such cases be given showing how the cost of power has been arrived at.

Naturally, when power is rented from a concern that makes the production of power a business, the publication of such data will be unnecessary.

It will probably be found upon investigation that, while the figures given may or may not be correct under conditions that pertain in some districts of the United States, they are not correct in other districts, and certainly are not correct in other countries.

Why did the authors ignore the cost of power when produced by internal combustion oil engines? By the use of these engines, *where local conditions are favorable,* it is claimed that power can be produced cheaper than by the use of producer gas or steam.

The question of the cost of producing power is one of great importance and one of which much has yet to be learned. The question is one particularly interesting to the electrochemist, because so many processes of great value are dependent for their commercial usefulness upon power being available at low cost.

The importance, therefore, of trustworthy data is very evident, and the Society would be doing work of world-wide importance if it could arrange for the compilation and publication of such data.

Robert M. Keeney (*Communicated*): I have read with interest the statement of Mr. Hansen regarding the manufacture of ferro-alloys and calcium carbide as a power consumer for power plants which need to tide over a few years of a poor market without too great an initial expenditure. This applies to the East as well as the West. While the raw ores would have to be shipped East or imported, the Eastern plant has the advantage of a closer market. On the other hand, the ores are found in the West close to cheap power, as, for example, in California.

Recently I have had occasion to inquire into power costs for an electric furnace plant for the manufacture of ferro-alloys, and find that there is at least one Eastern hydro-electric plant, outside of Niagara Falls, which will sell power for $27 per kilowatt year on a load factor of 85 percent, 24 hour power, 365 days per year.

There are several hydro-electric plants which have power for sale at a rate of 0.3 cents per kilowatt hour, or $27 per kilowatt year, on the basis of nine months guaranteed delivery, and one-quarter to one-third of the total amount delivered during the other three months. The period of low water comes for about a month in the winter and two months in the summer. As in

the case of several large French and Norwegian ferro-alloy and calcium carbide works which I have visited, the plant could be operated with a maximum of power during nine months and at a reduced rate for the remaining time. This practice is common in Europe. It works out well when the power company operates or owns an interest in the electric furnace plant, but complications might arise if an independent company was manufacturing. With power at these rates, and a fair contract, an independent company should be able to operate at a profit. One of the chief difficulties, from my experience, is to obtain a contract by which an independent company can be sure of a renewal of its contract at a reasonable rate after a period of, let us say, 10 years has elapsed. Most of these companies with surplus power for sale are located in regions where ordinary manufacturing will consume it in 20 to 30 years.

As Mr. Hansen states, ferro-silicon is a very great consumer of power. In the production of one long ton of 50 percent ferro-silicon, about 0.8 to 0.9 kilowatt year is consumed. The power cost, with power at $27 per kilowatt year, would be from 35 to 40 percent of the total cost when iron or steel turnings are used to supply the iron. If iron ore is used, the power consumption is about 20 percent greater than with turnings, in producing a 50 percent alloy. Ferro-chrome is not quite so great a consumer of power, since in the production of a short ton of 60 percent ferro-chrome, 0.7 kilowatt year is used. This amounts to about 25 percent of the total cost, with power at $27 per kilowatt year. Ferro-tungsten and the more costly alloys are consumed in such small quantities that they are not such great power consumers. A phase of electric furnace ferro-alloy production which appears to have a future is the production of ferro-manganese, which has been made chiefly in blast furnaces, because of the high loss by volatilization experienced by the first electric furnace manufacturers of ferro-alloys. Later work has shown it possible to so regulate the temperature of the electric furnace that comparatively little loss results. Some foreign works now produce ferro-manganese in the course of manufacture of silico-manganese. Calcium carbide is not as great a power consumer as ferro-silicon. European plants, where the calcium carbide is tapped from the furnace, use about 0.4 kilowatt year per short ton of

carbide. The power cost from their estimates would be about 25 to 30 percent of the total cost, with power at $27 per kilowatt year.

In 1912 I visited about thirty electrometallurgical plants in Europe, including ferro-alloy, calcium carbide, electric furnace steel, cyanamide, electric furnace zinc, aluminum and electric pig iron plants, for the purpose of studying these industries as power consumers. Of these, considering the individual power plant, and not involving great initial expenditure, the ferro-alloy and calcium carbide plants seemed most feasible, with possibly aluminum next, but these do not seem to be a safe investment under conditions existing in the United States for a power cost over $27 per kilowatt year, either to the power plant itself as a consumer, or to the independent company.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

ELECTRIC SMELTING OF CHROMIUM, TUNGSTEN, MOLYBDENUM AND VANADIUM ORES.

By Robert M. Keeney.*

INTRODUCTION.

Ores of chromium, tungsten, molybdenum and vanadium are found and mined throughout the western United States, but are not smelted in the locality of the mines. In the treatment of these ores the electric furnace has a field of its own for the production of ferro-alloys. At present the ores are shipped east or to Europe, after a concentration, for smelting into a metal or a ferro-alloy. In the United States some of the ores are treated by chemical and combustion furnace methods, as well as in the electric furnace, but in Europe, where the high temperature smelting of ores and the production of ferro-alloys has become a much greater industry than in the United States, practically all of the ores are treated in the electric furnace.

The experimental work contained in this paper was performed at the metallurgical laboratory of the Colorado School of Mines, under a Carnegie Research Scholarship granted by the Iron and Steel Institute of Great Britain. The sources of other information are the notes of a recent visit of the writer to electrometallurgical plants of Europe, and technical literature. This paper deals only with metallurgical reactions in smelting the ores mentioned, and not with the design or construction of electric furnaces for the performance of these reactions.

ELECTRIC SMELTING OF CHROMITE AND THE PRODUCTION OF FERROCHROME.

Historical.

With the exception of ferrosilicon, more ferrochrome is produced in the electric furnace than any other ferro-alloy manufac-

* Published by permission of the Director of the U. S. Bureau of Mines.

tured in this way. The alloy of iron and chromium called ferrochromium or ferrochrome has been known since 1820, through the studies of Faraday, Stodarts and Berthier. By 1886 there were several regular producers of ferrochrome, which was then made in the blast furnace or the crucible. In 1899 there began the production of ferrochrome in the electric furnace.

Moissan[1] reduced chromite ($FeO.Cr_2O_3$) with carbon as a reducing agent in the electric furnace and obtained a high-carbon ferrochrome. He also found lime to act as a decarburizing agent on a molten bath of ferrochrome under the arc of an electric furnace. A low-carbon product resulted.

Neumann[2] investigated the use of ferrosilicon as a reducing agent in the electric furnace in an effort to produce a carbon-free alloy. The resulting alloy was high in silicon, and also contained considerable carbon from the ferrosilicon used as a reducing agent.

Theoretical Considerations.

Reduction with Carbon.—In the reduction of chromium from chromite to form ferrochrome with the iron reduced, reduction of chromic acid begins at 1185° C.[3] Reduction takes place according to the formula:

$$FeO.Cr_2O_3 + 4C = FeCr_2 + 4CO$$

A pure chromite ore contains 68 percent Cr_2O_3 and 32 percent FeO. For reduction of chromite 30 parts of pure carbon are theoretically necessary for every 100 parts of iron and chromium reduced. In practice this amount is exceeded considerably.

In ferrochrome manufacture practically all of the reduction is performed by solid carbon and not by carbon monoxide gas. On the basis of the carbide $Fe_3C.3Cr_3C_2$ forming in reduction of chromite with carbon, as has been found to be the case, the reduction formula is as follows:

$$9FeO.9Cr_2O_3 + 50C = [2(Fe_3C.3Cr_3C_2) + 3Fe] + 36CO$$

The ferrochrome resulting would contain theoretically 10.4 percent carbon, 31.4 percent iron and 58.2 percent chromium. For

[1] The Electric Furnace, p. 152.

[2] Stahl und Eisen, p. 356 (1908).

[3] Transactions Chemical Society (England), 93, 1484 (1908); Electrochemical and Metallurgical Industry, 7, 119 (1909).

reduction of 100 parts of this alloy 37 parts of carbon are necessary, which explains the difference between the theoretical and practical amount of carbon necessary, if calculation is made without considering the formation of the double carbide.

Reduction with Silicon and Other Reducing Agents.—Several United States patents have been issued for the reduction of metallic oxides by the use of silicon as a reducing agent. The action takes place according to the following formula:

$$2FeO.3Cr_2O_3 + 4Si = 2FeCr_2 + 4SiO_2$$

Thus for the reduction of 100 parts of ferrochrome containing 65 percent chromium, 35 parts of silicon are necessary. In patents granted to F. M. Becket,[4] based on this reaction, it is claimed that a ferrochrome very low in silicon can be made by this method, although this is not shown in the researches of Neumann, where the silicon percentage of the alloy was high. Patents based upon the same reaction, using ferrosilicon as a reducing agent, have been granted to E. F. Price.[5] These processes are expensive, and the silicon in the alloy tends to be high, and as a result these methods are not widely used in actual manufacture.

F. M. Becket[6] proposes the use of calcium carbide as a reducing agent. He claims that the calcium reduces the metals, and that the carbon oxidizes or remains with the resulting alloy.

$$3FeO.3Cr_2O_3 + 4CaC_2 = 3FeCr_2 + 4CaO + 8CO$$

This method has no commercial application of great extent because of the expense. For the reduction of 100 parts of 65 percent ferrochrome, 53 parts of calcium carbide are necessary.

Rossi,[7] using aluminum as a reducing agent in the electric furnace, has produced ferrochrome containing 68.24 percent chromium, 1.85 percent silicon, 1.0 percent carbon, 0.5 percent aluminum from chromite containing 50.29 percent Cr_2O_3 and 16.01 percent FeO. This also is an expensive method because of the expense of the aluminum. Also, as the presence of

[4] U. S. Patent No. 854,018, May 21, 1907; U. S. Patent No. 866,561, September 17, 1907; U. S. Patent No. 891,898, June 30, 1908.

[5] U. S. Patent No. 865,609, September 10, 1907; U. S. Patent No. 862,996, August 13, 1907; U. S. Patent No. 852,347, April 30, 1907.

[6] U. S. Patent No. 898,173, September 8, 1908.

[7] Mineral Industry, p. 693 (1903).

aluminum in an alloy is more objectionable to many steel makers than carbon, no advantage is gained in this respect.

General Manufacture.

Ferrochrome, up until the introduction of the electric furnace, in 1899, was manufactured entirely in either the crucible or blast furnace. The crucible was used only for the production of the higher grades, and the charge consisted of the chromite, charcoal and a flux of lime, borax, fluorspar or water glass. In the blast furnace chromite is reduced with some difficulty with the high fuel consumption of about three tons of coke to one ton of ferrochrome produced. Theoretically it should be possible to get a ferrochrome containing 65 percent chromium from an ore containing 60 percent Cr_2O_3 and 20 percent FeO, but generally a ferrochrome containing from 30 to 40 percent chromium and 6 to 12 percent carbon is the product in the blast furnace. Very little ferrochrome is now made by either the crucible or blast furnace processes, because it can be made cheaper in the electric furnace at points where electric power is cheap. A small amount of carbon-free ferrochrome is now made by the thermit process.

Ores and Raw Materials.

The greater part of the ores now used in the electric furnace manufacture of ferrochrome come from Turkey, New Caledonia, Cuba and New South Wales. There are considerable deposits of chromite ore in Canada and California, but they are so far from the market that the production does not amount to much.

The foreign manufacturers as a rule use only chromite containing less than 0.1 percent phosphorus and sulphur and about 50 percent Cr_2O_3. They usually use no lime in the charge, as it has been found preferable by them to use pure raw materials and keep the amount of slag as low as possible.

In most cases high-grade anthracite coal is the reducing agent. The only American manufacturers of ferrochrome, according to their patent specifications, run with a lime slag. They sometimes use Cuban ore, which contains considerable phosphorus and sulphur. Coke or charcoal may be used instead of anthracite coal, but the latter is better, as it may be obtained in a more finely pulverized state, which gives a more intimate contact with the

chromite and consequently a better reduction. As electric furnaces do not have a high shaft, it is not necessary to use a reducing material of high compressive strength.

Furnace.

The electric furnace used in the manufacture of ferrochrome may be one of several types, all of the principle of the Siemens crucible with a conducting hearth, or similar to the Héroult steel furnace, with vertical electrodes in series. In a conducting hearth furnace the lining of the walls may be of chromite or more commonly magnesite or dolomite tar mixture, with a carbon block in the bottom. Or the construction of the Girod furnace may be used, in which a conducting hearth of iron is buried in the refractory bottom; or the "series" furnace construction may be employed, with a non-conducting hearth in which the lining of hearth and walls is entirely of a non-carbon refractory material, such as dolomite, magnesite or chromite. The walls are thin, not over 9 inches thick, but the electrodes are far enough from them to allow freezing of the charge on the walls, thus forming a very refractory lining. Some furnaces were water-cooled at first to assist this, but it is not generally done any longer. Ferrochrome freezes to a certain extent on the bottom, so that molten metal does not have much chance to come in contact with the carbon bottom.

Process of Manufacture.

The process of manufacture of ferrochrome consists of mixing a charge of chromite and anthracite coal in proportions based upon the theoretical calculations given. This is shoveled into the open top of the furnace around the electrodes. The furnace is charged at regular intervals so as to keep the top in a solid condition. The ore is very finely powdered, but the coal is about as it comes from the mine. There is some loss by ore being carried off with the gases which escape at the top of the furnace.

The ferrochrome is tapped off from the bottom, at intervals of from two to four hours, into iron pots set on cars, from which it and any slag are dumped when solid. The slag and metal come out of the same tap hole. When cold the metal is broken up with hammers to separate it from the slag and packed into kegs or

boxes. For tapping an iron rod is used to open up the tap hole, which is plugged with fire clay.

Ferrochrome manufacture is carried on continuously, in some cases for two years, before shutting down the furnace for repairs. In one operation, directly from ore, ferrochrome containing down to 5 percent carbon can be made economically. It has been possible to produce an alloy containing as low as 2 percent carbon in one operation, but great care is necessary in the regulation of the charge and the operation of the furnace. The slags from the average ferrochrome furnace making a 5 percent carbon alloy run from 0.5 to 1.0 percent Cr_2O_3.

Refining of Ferrochrome.

Decarburization of high-carbon ferrochrome is accomplished by melting the alloy with a slag of lime, chromite ore and a little fluorspar, according to the following reaction:

$$2[2(Fe_3C.3Cr_3C_2) + 3Fe] + 7FeO.Cr_2O_3 = 25FeCr_2 + 28CO$$

For each 100 parts of completely decarburized alloy, 39 parts of pure chromite ore are necessary, or for each 100 parts of ferrochrome containing 10.6 percent carbon, 31.3 percent iron and 58.11 percent chromium, 36 parts of pure chromite ore are necessary for complete decarburization. The resulting alloy would contain 66.6 percent chromium and 33.3 percent iron. In practice the lime is added simply to flux any silica in the ore, to assist in elimination of silicon in the ferrochrome and cause possible removal of carbon or calcium carbide, as the operation is performed in an arc furnace. The fluorspar makes the slag more fluid. Gin[8] claims that a high temperature is not necessary for decarburizing, but this is not borne out in practice.

E. F. Price[9] proposes to decarburize ferrochrome by passing an oxidizing gas through the molten metal, but the process is not used in practice.

F M. Becket[10] proposes to make a ferrochrome high in silicon, containing approximately 51.3 percent chromium, 17.5 percent iron, 30.0 percent silicon and 1.2 percent carbon in the first stage, and then remove the silicon by adding chromite in the propor-

[8] Trans. Am. Electrochem. Soc., **15**, 225 (1909).
[9] U. S. Patent No. 886,858, May 5, 1908.
[10] U. S. Patent No. 891,898, June 30, 1908.

tion of six parts of chromite to one part of silicon in the alloy, this being slightly in excess of the amount theoretically necessary to oxidize all the silicon to silica. He gets an alloy containing 70 percent chromium, 28.9 percent iron, 1.0 percent carbon and 0.10 percent silicon. The necessity of such a refining process shows the weakness of methods using ferrosilicon or silicon as a reducing agent in that the percentage of silicon in the alloy can not be kept low in one operation without high losses of chromium in the slag.

The refining of the high-carbon grades of ferrochrome is carried on in an arc furnace, where the alloy is subjected to a prolonged heating with a slag of chromite, lime and fluorspar, in proportion to the amount of carbon and silicon to be removed and the silica which must be fluxed in the chromite used. The ferrochrome covered by this slag is heated at a high temperature for a length of time dependent upon the carbon in the alloy. The resulting slag contains about 25 percent Cr_2O_3, and is ground up to send through the smelting furnace again, so that there is not much loss of chromium. By this method it is possible to reduce the carbon from 10 percent to 0.25 percent, but the more usual low-carbon grade contains 0.5 percent carbon.

The percentage of chromium in the ferrochrome remains about the same as the carbon is decreased from 9.5 to 0.6 percent, the iron increasing with the decrease of carbon. This would seem to show that, in refining, the iron oxide of the chromite is first reduced, the chromic oxide merely serving to keep a slag with an excess of chromium so as to prevent any final lowering of the percentage of chromium in the alloy. As the carbon is oxidized, so is the silicon, down to 0.2 percent, from 2 to 5 percent in the crude alloy. Aluminum is also considerably eliminated, and phosphorus and sulphur are reduced to a slight degree.

Although the market is small for very low-carbon ferrochrome, the difference in price between the alloy with 9 percent carbon and the 0.5 percent product does not seem warranted. The refining expense is almost entirely in power, and with cheap power, as is generally the case at a ferro-alloy plant, the expense is not as high as $300 per ton, the difference in price of the products mentioned.

Processes which have been tried out, based upon the use of silicon or some reducing agent other than carbon, have not been commercially successful because of the much higher cost of such reducing materials as silicon, ferrosilicon and aluminum over that of carbon. Also the products may be contaminated with the reducing material, just as when carbon is used, so that a subsequent refining is necessary. Carbon-free ferrochrome for high-grade steels has, however, been successfully made by the thermit process, but the amount made in this way is comparatively small.

Experimental Smelting of Chromite in the Electric Furnace.

Some experiments were made by the writer in 1912 on the production of ferrochrome from chromite with carbon as a reducing agent. The experiments were performed in a furnace lined with magnesite, having one upper graphite electrode and a conducting hearth of iron rods imbedded in magnesite. The interior dimensions of the crucible were 4 inches wide, 8 inches long and 8 inches deep (10 x 20 x 20 cm.). This furnace took from 8 to 12 kilowatts of single-phase current at from 30 to 50 volts, and held from 12 to 15 pounds (6 kg.) of the cold charge.

The chromite used, Table I, was a foreign grade used commonly for furnace linings. Analyses of the coke, lime and fluorspar used are also given in Table I.

TABLE I.

Raw Materials.

Chromite		Coke		Lime		Fluorspar	
	Percent		Percent		Percent		Percent
Cr_2O_3	46.35 (31.7 Cr.)	Fixed carbon	81.48	CaO	92.76	CaF_2	90.00
FeO	21.45 (16.6 Fe.)	Volatile and combustible	0.25	SiO_2	1.34	$CaCO_3$	0.80
SiO_2	5.48	Ash	17.92	$Al_2O_3 + Fe_2O_3$	1.90	Al_2O_3	8.50
Al_2O_3	12.80	Moisture	0.35	MgO	Trace		
MgO	10.08	FeO	1.20	Phosphorus	0.06		
CaO	Trace	SiO_2	13.62				
Phosphorus	0.013	CaO	0.85				
Sulphur	0.45	Phosphorus	0.08				
		Sulphur	0.54				

In these experiments, contrary to the usual European practice in ferrochrome manufacture, lime was used as a flux to note its desulphurizing and dephosphorizing effect with an ore not so pure as the usual ore used in ferrochrome production. In usual practice only the most pure raw materials available are charged so as to avoid the excess slag caused by adding lime. The only slag formed is from the coal or coke and chromite.

Five experiments were made in which the furnace was charged and tapped at the completion of reduction, Table II. In these experiments the chromium percentage of the alloy seemed to run considerably lower than the theoretical calculation, although this is accounted for in a measure by the fact that the carbon in the metal was not considered in calculating the theoretical percentage.

The carbon was high in all of the alloys, and does not seem to be influenced by the carbon in the charge as long as there is excess, which was the case. Phosphorus was high in all of the products. Apparently all of the phosphorus charged was concentrated in the metal. This was caused by the excessive reducing conditions, which did not permit the slagging of the phosphorus as calcium phosphate. The ore contained considerable sulphur (0.45 percent), but almost all of this passed into the slag, probably as calcium sulphide. There was considerable loss of chromium and iron in the slag. In some cases the percentage of iron in the slag exceeded that of the chromium, but in no case did the percentage of chromic oxide in the slag exceed 9 percent, or 4.69 percent chromium. This shows that, in the endeavor to keep the carbon in the alloy low, the amount of coke added was not sufficiently in excess for complete reduction, or that the furnace had not been hot enough. The former was probably the case, as the furnace was operated as an arc furnace throughout the experiments.

Two experiments, Nos. 6 and 7, were made in which the furnace was left full and several tappings made during the run. In this way the usual practice of ferrochrome manufacture was followed. Commercial ferrochrome is in grades of from 0.5 to

Table II.

Production of Ferrochrome Directly from Ore.

Experiment No.	1	2	3	4	5	6						7		
						a	b	c	d	e	f	a	b	c
Charge:														
Chromite, pounds	10.00	10.00	10.00	10.00	10.00	.....	.....	.....	60.00	.....	.....	.....	40.00	.....
Coke, "	1.87	1.87	1.87	1.63	1.36	.....	.....	.....	11.23	.....	.....	.....	8.10	.....
Lime, "	1.25	1.25	1.25	1.25	1.25	.....	.....	.....	6.70	.....	.....	.....	4.50	.....
Fluorspar, "	0.12	0.12	0.12	0.12	0.12	.....	.....	.....	0.68	.....	.....	.....	0.44	.....
Product, alloy:														
Chromium, percent	51.80	48.60	49.70	52.70	57.10	50.69	68.20	58.90	56.10	68.00	67.10	62.60	72.70	68.00
Carbon, "	10.05	8.74	11.50	3.72	9.28	6.50	9.31	8.72	7.89	4.32	7.40	5.28	8.32	4.43
Silicon, "	0.81	1.10	1.26	0.87	0.35	0.67	0.60	1.23	1.25	0.96	1.03	0.79	0.56	0.94
Phosphorus, "	0.128	0.133	0.110	0.106	0.120	0.106	0.099	0.108	0.119	0.106	0.106	0.121	0.119	0.104
Sulphur, "	0.073	0.038	0.096	0.061	0.085	0.071	0.105	0.055	0.016	0.031	0.054	0.020	0.023	0.027
Slag, Cr_2O_3, "	8.03	7.57	6.96	8.98	9.81	9.15	12.60	10.72	13.43	13.13	13.34	8.78	11.40	11.35
" SiO_2 "	23.40	24.60	23.40	20.25	26.60	32.60	34.20	29.20	28.00	28.90	28.00	36.84	30.50	29.40
" FeO, "	9.02	7.29	18.96	15.10	10.17	6.10	14.82	15.43	14.81	15.17	17.55	8.73	16.99	18.10
Calculated percentage chromium	65.50	65.50	65.50	65.50	65.50	.....	.....	.....	65.50	.....	.....	.....	65.50	.....
Pounds carbon per lb. iron and chromium	0.312	0.312	0.312	0.271	0.228	.....	.....	.....	0.312	.....	.....	.....	0.418	.....
Calculated weight, lbs.	4.45	4.00	4.47	3.87	5.06	.....	.....	.....	29.3	.....	.....	.....	19.4	.....
Ferrochrome tapped, lbs	3.5	4.87	5.15	2.44	2.56	2.66	2.13	3.21	3.21	3.21	3.50	1.50	2.51	7.00
Percent tapped	78.5	89.9	86.3	62.95	53.30	Total tapped, 17.92 lbs., 61.8%						11.1 lbs., 66.8%		
Electrode consumption per ton (2,000 lbs.) tapped, pounds	210	150	142	204	189	.....	.....	.....	48.1	.....	.....	.....	16.3	.....
Kilowatt hours per lb. alloy tapped	3.18	2.73	2.57	6.38	4.71	.....	.....	.....	3.02	.....	.....	.....	3.53	.....
Kilowatt years per ton (2,000 lbs.) alloy tapped	0.73	0.62	0.59	1.46	1.08	.....	.....	.....	0.69	.....	.....	.....	0.81	.....

10 percent carbon, containing 60 to 65 percent chromium. In these experiments the percentage of chromium in the alloy closely approached the theoretical. The percentage of carbon was also lower in these alloys because of the decarburizing effect of the unreduced oxide in the charge above the molten metal. The slag loss of iron and chromium was higher in these runs than in the intermittent experiments because of unreduced oxide getting into it on tapping.

A total of 50 pounds of ferrochrome was tapped during the experiments, or an average extraction of 69.5 percent. The average iron content of the slag for all experiments was 13.4 percent FeO (10.4 percent Fe); the average chromium in the slag was 10.3 percent Cr_2O_3 (6.6 percent Cr). In the intermittent experiments the average percentages in the slag were 12.1 percent FeO (9.4 percent Fe) and 8.07 percent Cr_2O_3 (5.32 percent Cr). Thus, of the loss of 30.5 percent which was not tapped, 11.9 percent was chromium and 18.6 percent was iron, figuring that of the total average loss 39 percent was chromium and 61 percent iron. A large part of the ferrochrome not tapped stuck in the furnace. While the average electrode consumption was 158 pounds per ton of 2,000 pounds, the figure obtained in experiment No. 6 would probably be obtained in practical operation (48.1 pounds). The average power consumption was 3.7 kilowatt hours per pound, or 0.85 kilowatt year per ton.

The conclusions drawn are: First, that ferrochrome can be easily manufactured directly from chromite in the electric furnace; second, that the percentage of carbon in the ferrochrome can not be kept low by regulating the carbon charged without excessive loss of chromium in the slag; third, that the percentage of carbon in the ferrochrome must be regulated by decarburization with an oxide slag of iron or chromite after tapping off the slag from reduction; fourth, that silicon and phosphorus can not be kept low in the alloy under the strong reducing conditions necessary; fifth, that sulphur can be easily slagged; sixth, that, as the addition of lime does not seem to aid in removing phosphorus, it is advisable to use no lime, but as pure raw materials as possible; and, seventh, that the power consumption should not

exceed 3.7 kilowatt hours per pound of ferrochrome tapped, or 0.85 kilowatt year per ton.

Power and Electrode Consumption.

The power consumption in a 750-kilowatt ferrochrome furnace of the Alby carbide type at the plant of the Meraker Electric Smelting Company, at Kopperaaen, Norway, was recently given as 3.0 kilowatt hours per pound, or 0.68 kilowatt year per short ton in making a 5 percent carbon and 65 percent chromium ferrochrome. At Kanawha Falls,[11] Virginia, the Electrometallurgical Co. made ferrochrome in an arc crucible electric furnace with a power expenditure of 3.6 kilowatt hours per pound, or 0.72 kilowatt year per ton. This product contained 70.96 percent chromium, 23.23 percent iron, 5.21 percent carbon, 0.5 percent silicon, 0.008 percent phosphorus and 0.078 percent sulphur. At both Meraker and Kanawha Falls an ore containing about 50 percent Cr_2O_3 was used. In the experiments of the writer, which have been described, a product containing from 50 to 68 percent chromium and from 4.32 to 9.31 percent carbon was obtained, using a 46.35 percent Cr_2O_3 ore with 3.02 kilowatt hours per pound, or 0.69 kilowatt year per ton.

The electrode consumption in ferrochrome manufacture depends somewhat upon the degree of carbon in the ferro-alloy being produced, as if the carbon of the charge is materially reduced, the electrode will be oxidized by the charge. The consumption varies from 50 to 100 pounds per short ton of ferrochrome produced with an open-top furnace. As most of the ferrochrome furnaces do not use continuously feeding electrodes, this item will probably be kept at a much lower figure in the future. At present in many plants from one-half to one-third of the electrode is not used, and, unless the works has its own electrode plant where stumps can be made into new electrodes, this is lost so far as its electrode value is concerned.

Aside from the electrode item, the repair and upkeep cost on a ferrochrome furnace is not high. The linings last from one to three years without being repaired.

[11] Electrochemical Industry, Nov., 1904, p. 450.

ELECTRIC SMELTING OF TUNGSTEN ORES AND THE PRODUCTION OF FERROTUNGSTEN.

Historical.

Ferrotungsten of low-percentage tungsten was made by Berthier in 1834. Caron studied iron tungsten alloys in 1868, and determined that the hardness increased with the percentage of tungsten. In 1866 Biermann made some ferrotungsten from tungsten trioxide and iron in the crucible. This method was used up to the introduction of the electric furnace in 1900, since when most of the ferrotungsten has been made in the electric furnace, although some is still manufactured in small quantities, containing less than 60 percent tungsten, in the crucible. The crucible product is made by reduction of tungsten trioxide with carbon. In electric furnace methods raw ores or concentrates are reduced.

Stassano[12] conducted experiments on the manufacture of ferrotungsten from wolframite concentrates in the electric furnace, using charcoal as a reducing agent. A high-grade product containing from 58 to 70 percent tungsten and about 2.5 percent carbon was produced. The average energy consumption was 3 kilowatt hours per pound of alloy produced. A flux of lime was used, and the ore briquetted.

Theoretical Considerations.

Ferrotungsten can be made by reduction of wolframite, ferberite or scheelite with aluminum, silicon or carbon. Rossi[13] reduced ferberite with aluminum in a Siemens type of electric furnace. An alloy containing 75.9 percent tungsten, 21.4 percent iron, 1.6 percent silicon, 0.08 percent sulphur and 0.9 percent carbon was made from concentrates containing 69.8 percent WO_3, 20.25 percent FeO and 5.04 percent SiO_2. The reaction used is as follows:

$$3Fe_2WO_4 + 8Al = 3Fe_2W + 4Al_2O_3$$

For the reduction of 100 parts of 62.3 percent tungsten ferrotungsten from 122 parts of ferberite, 24.2 parts of aluminum are necessary.

[12] Electrochemical and Metallurgical Industry, 6, 315 (1908).
[13] Mineral Industry, p. 693 (1903).

Gin has produced ferrotungsten by the reduction of scheelite with a bath of 20 percent ferrosilicon in the electric furnace according to the following reaction:

$$3CaWO_4 + 4Fe_2Si = (2Fe_2W + Fe_3W) + 3CaSiO_3 + FeSiO_3$$

The most common method for the production of ferrotungsten is by the reduction of ferberite, wolframite or scheelite concentrates in the electric furnace with carbon as a reducing agent, followed by a subsequent decarburization and refining. Wolframite or ferberite are readily reduced in this manner, but there is difficulty in the reduction of scheelite. Ferberite is wolframite, $(Fe.Mn)WO_4$, in which the manganese is replaced by iron so as to give $(Fe.Fe)WO_4$. Reduction occurs with carbon according to the following reaction:

$$Fe_2WO_4 + 4O = Fe_2W + 4CO$$

Theoretically the product contains 62.3 percent tungsten. For the reduction of 100 parts of this alloy from 122 parts of ferberite, 16.2 parts of carbon are necessary.

Scheelite[14] is now smelted by one company with sulphide of iron and carbon in the electric furnace. Lime is also added to flux the silica. The following reaction takes place:

$$CaWO_4 + FeS + 4C = FeW + CaS + 4CO$$

The product is said to be a ferrotungsten containing little carbon.

After much experimental work on a large scale, it seems that reduction with carbon followed by decarburization with iron ore or tungsten concentrates is the cheapest and most efficient method of making ferrotungsten in the electric furnace. Aluminum is expensive, and goes into the alloy to a certain extent, as also does silicon.

Ferrotungsten may be decarburized[15] by covering the fused metal in the electric furnace with hammer scale, when the following reaction takes place:

$$Fe_6O_7 + 7W_2C = 6FeW_2 + W_2 + 7CO$$

[14] Engineering and Mining Journal, **93**, 173 (1912).
[15] Trans. Am. Electrochem. Soc., **13**, 481 (1908).

The reaction is not strictly quantitative, since there is always formed some ferrous tungstate.

$$Fe_6O_7 + 3W_2C = 5FeW + FeWO_4 + 3CO$$

By this reduction it is possible to make ferrotungsten containing 80 percent tungsten and less than 0.5 percent carbon. Ferberite or wolframite may be used instead of hammer scale. Slags high in tungsten are run through the reducing furnace again. By the above method carbon can not be reduced below 0.3 percent in the alloy.

Ferrotungsten with less than 80 percent tungsten can be made by adding metallic iron to the bath and decarburizing with iron oxide and hammer scale. This method yields alloys with as low as 0.15 percent carbon. Decarburizing with iron oxide has a tendency to cause loss of tungsten in the slag by the formation of tungstate of iron. This may be reduced somewhat by making the decarburizing slag rather acid, when ferrous silicate is formed.

Process of Manufacture.

Ferrotungsten is manufactured from ores in three ways: First, by direct reduction with carbon in a crucible; second, by reduction in an electric furnace by some reducing agent other than carbon; and, third, by direct reduction with carbon in an electric furnace.

In manufacturing ferrotungsten by the crucible process,[16] concentrates are placed in a clay-lined crucible with the proper proportions of reducing agent and flux, and heated to a high temperature in a gas-fired furnace. There is considerable wear on the crucible in this method. For a 30-percent tungsten alloy the crucible will last about three heats, but for a 65 to 75-percent product they last but one heat. Higher percentage tungsten alloys than this are not made in the crucible furnace.

In the electric furnace process, ferberite, wolframite, hubnerite or scheelite are reduced with carbon in an arc furnace. As with many expensive alloys, the process is generally an intermittent one, *i. e.,* the reduced charge is tapped from the furnace before another charge is added, or the charge is let solidify and chiseled out of the furnace. Sometimes a campaign of several days is

[16] Engineering and Mining Journal, Nov. 12, 1910, p. 959.

made. The reduced alloy is either decarburized with refining slags in the same furnace or melted and refined later in another furnace. The furnaces used are similar in design to the ones described for ferrochrome production, but a more complete recovery is effected by the use of a tilting rather than a stationary furnace. Wolframite, ferberite and scheelite are reduced easily in the electric furnace, most of the manganese being volatilized. Scheelite is more difficult to reduce, and very sticky basic slags result. There is greater loss in its reduction, so that it does not command as high a price as the other ores, selling for about $1 per unit less. The product is tapped into molds, broken up on cooling, and packed into kegs or boxes.

The largest producers of tungsten ores are Australia, Colorado, California and Argentine. The standard concentrate contains 60 percent tungsten trioxide, WO_3. The price varies from $5 to $8 per unit in the 60 percent grade. That is, if the rate was $5, a 60 percent concentrate would be worth $300 per short ton. A typical Colorado ore, ferberite, is that given under the writer's experiments. A typical scheelite contains 69.50 percent WO_3, 16.04 percent CaO, 0.54 percent FeO, 0.18 percent MnO, 0.14 percent P_2O_5 and 12.46 percent SiO_2.

Experimental Electric Smelting of Colorado Ferberite.

In 1911 the writer performed some experiments upon the reduction of ferberite with carbon to produce ferrotungsten in the electric furnace. The furnace was that described under the production of ferrochrome. Analyses of the coke and lime employed are given in Table I. Pure calcium fluoride was used. The iron ore contained 94.9 percent Fe_2O_3, 4.10 percent SiO_2, 0.79 percent CaO, 1.46 percent Al_2O_3, 0.05 percent phosphorus and 0.03 percent sulphur. Ferberite concentrates from Boulder County, Colorado, were obtained of the following composition: 58.72 percent WO_3 (46.6 percent tungsten), 30.08 percent FeO (23.4 percent iron), 2.55 percent MnO (0.84 percent manganese), 4.86 percent SiO_2, 3.46 percent CaO, 0.34 percent phosphorus and 0.20 percent sulphur.

In each experiment a decarburizing slag of iron ore, lime and fluorspar was added to the furnace when reduction of the charge

was completed, and allowed to act for ten to twenty minutes. It was tapped from the furnace with the ferrotungsten and the slag from reduction.

As shown in Table III, the alloys obtained, with two exceptions, run somewhat lower in tungsten than was calculated. This was caused by three conditions: First, the presence of iron in the furnace from a previous run with iron ore; second, loss of tungsten in the slag, and, third, the iron reduced from the decarburizing slag. The carbon of all the alloys was low for ferrotungsten. It was evident that this was assisted considerably by

TABLE III.

Production of Ferrotungsten Directly from Ore.

Experiment No.	1	2	3	4	5	6	7
Charge:							
Ferberite, pounds	5.00	10.00	5.00	5.00	6.00	6.00	6.00
Hematite, "	1.50	3.00	1.50	1.50	.....	.....	.....
Coke, "	1.25	3.00	1.37	1.52	1.12	1.12	1.12
Lime, "	0.75	1.56	0.90	0.95	0.75	0.75	0.75
Fluorspar, "	0.062	1.24	0.062	0.062	0.062	0.062	0.062
Refining slag:							
Hematite, "	0.75	0.75	0.75	0.75	0.75	0.75	0.75
Lime, "	0.20	0.20	0.20	0.20	0.20	0.20	0.20
Fluorspar, "	0.044	0.044	0.044	0.044	0.044	0.044	0.044
Product:							
Alloy, Tungsten, percent	38.74	49.64	41.39	43.35	42.10	37.50	59.01
" Carbon, "	1.77	1.82	2.05	2.51	1.66	2.54	2.02
" Manganese, "	0.32	0.16	0.42	0.38	0.16	0.33	0.16
" Silicon, "	0.47	0.31	0.24	0.44	0.14	0.07	0.09
" Phosphorus, "	0.044	0.046	0.042	0.048	0.051	0.054	0.020
" Sulphur, "	0.032	0.068	0.089	0.017	0.014	0.021	0.015
Slag, WO_3 "	8.64	4.14	7.20	4.24	4.00	5.28	4.88
" SiO_2, "	30.12	31.10	31.00	31.40	30.40	29.28	29.64
" CaO, "	21.30	37.00	30.00	24.60	28.00	21.10	29.30
" MgO, "	16.60	12.00	19.60	26.64	24.48	23.64	18.80
" FeO, "	12.60	12.00	5.84	4.52	5.84	5.10	6.87
" Al_2O_3, "	12.10	5.70	5.24	6.95	6.22	11.67	7.88
Calculated percent W in product	50.00	50.00	50.00	50.00	65.00	65.00	65.00
Pounds carbon per lb. W and Fe	0.202	0.222	0.222	0.212	0.214	0.214	0.214
Calculated alloy, pounds	5.08	10.16	5.08	5.08	4.30	4.30	4.30
Alloy tapped, pounds	2.00	8.78	5.62	2.20	6.50	2.20	4.62
Kilowatt hours per lb. tapped	4.76	2.63	2.00	5.78	1.86	5.25	1.96
Kilowatt years per ton tapped	1.09	0.60	0.46	1.32	0.42	1.20	0.45

the use of the decarburizing slag of iron oxide and lime, and that with a longer decarburization period the carbon in the alloy could be reduced still further. The greater part of the manganese in the charge was either slagged or volatilized. The percentage of silicon was high in some of the products, but much lower when there was no iron ore in the charge. Phosphorus and sulphur were easily slagged in spite of a high percentage of both in the ore. The slags contained from 4.66 to 8.64 percent tungsten oxide, or 3.7 to 6.8 percent tungsten, and ferrous oxide in about the same percentage.

The commercial electric furnace ferrotungsten contains from 50 to 80 percent tungsten and 0.5 to 4 percent carbon.

During the experiments 23 pounds of ferrotungsten were made, or an extraction of 83.8 percent. The electrode consumption for the two experiments in which it was determined averaged 150 pounds per ton. This figure would be considerably reduced in large-scale operations. The average energy consumption for the seven experiments was 3.46 kilowatt hours per pound tapped, or 0.79 kilowatt year per ton. The ferrotungsten was hard and brittle. Castings were free from blow holes.

Conclusions drawn are: First, ferrotungsten can be produced directly from ferberite in the electric furnace; second, by the use of a decarburizing slag before tapping, the percentage of carbon in the alloy can be kept below 2 percent; third, manganese, silicon, phosphorus and sulphur do not enter the ferro-alloy in high percentages; fourth, the loss of tungsten in the slag need not be excessive, and, fifth, the power consumption need not exceed 3.46 kilowatt hours per pound of ferrotungsten tapped, or 0.79 kilowatt year per ton.

ELECTRIC SMELTING OF MOLYBDENITE AND THE PRODUCTION OF FERROMOLYBDENUM.

Historical.

Ferromolybdenum is generally manufactured in the electric furnace from raw sulphide ore of molybdenum, molybdenite. It is also made by reduction of the roasted sulphide with carbon in a crucible or an electric furnace. The alloy is not widely used because of its high cost, which is caused by the irregularity of the

ore supply. With a better and steadier source of ore, ferromolybdenum would probably be widely used in place of ferrotungsten. Ferromolybdenum was made from roasted ore in the crucible before the introduction of the electric furnace, but has been produced commercially directly from the sulphide only since the electric furnace has been used, 1900.

Guichard[17] obtained a crude molybdenum from natural molybdenite by reduction with carbon in the electric furnace. The product was really a carbide produced according to the reaction:

$$2MoS_2 + 3C = Mo_2C + 2CS_2$$

Lehner[18] produced ferromolybdenum from molybdenite in the electric furnace, using lime as a desulphurizing agent and carbon as a reducing agent. A ferromolybdenum of low sulphur content was produced. This reduction was according to the reaction:

$$MoS_2 + 2CaO + 2C = Mo + 2CaS + 2CO$$

Neumann[19] attempted reduction of molybdenite according to the reaction:

$$MoS_2 + Si = Mo + SiS_2$$

but obtained poor results.

Theoretical Considerations.

A commonly used basis for figuring the charge for reduction of ferromolybdenum from molybdenite is according to the reaction used by Becket in his patent,[20] where carbon is the reducing agent and lime a desulphurizing agent.

$$2MoS_2 + 2CaO + 3C = 2Mo + 2CaS + 2CO + CS_2$$

Pure molybdenite contains 60 percent molybdenum and 40 percent sulphur. According to the above reaction, 100 parts of molybdenum are reduced from 170 parts of molybdenite by 18.8 parts of carbon. To complete the reaction, for every 100 parts of molybdenum present as the molybdenite, 58 parts of lime are

[17] Comptes Rendus, **122**, 1270.
[18] Metallurgie, **3**, 549 (1906).
[19] Stahl und Eisen, **28**, 356 (1908).
[20] U. S. Patent No. 835,052, Nov. 6, 1906.

necessary for slagging of the sulphur. Using calcium carbonate instead of lime, the reaction is:

$$2MoS_2 + 2CaCO_3 + 5C = 2Mo + 2CaS + CS_2 + 6CO$$

Using calcium carbide as a reducing agent, the following reaction takes place:

$$5MoS_2 + 2CaC_2 = 5Mo + 2CaS + 4CS_2$$

Becket in his patent states that an alloy containing less than 0.2 percent carbon can be made by this method. Molybdenum does not combine as readily with carbon to form carbides as some of the other metals, such as chromium and tungsten.

A patent was granted Becket,[21] based upon the desulphurizing of molybdenite with silica:

$$MoS_2 + Si = Mo + SiS_2$$

From the experiments of Neumann this does not appear to be a very satisfactory method. By this method the production of 100 parts of molybdenum requires 29 parts of silicon, which would probably be used in the form of ferrosilicon, the iron in it making the desired percentage in the ferromolybdenum.

Process of Manufacture.

The commercial manufacture of ferromolybdenum is carried on in an electric furnace of the electrode type operated as a resistance furnace, or in a crucible furnace of the resistance type. Comparatively little ferromolybdenum is made because of difficulty in getting ores. This is due to the fact that satisfactory methods of concentration for molybdenite ores have not been fully worked out as yet. There are deposits of molybdenite in the United States in Maine, Oregon, Colorado, Nevada and Washington, also in Canada, Germany, Japan, Mexico, New South Wales, New Zealand, Norway, Peru and Queensland. Queensland and New South Wales are the largest producers.

Buyers require an ore or concentrate containing 90 to 95 percent molybdenite, for which the price is about $450 per short ton.

In commercial manufacture, raw or roasted molybdenite, iron

[21] U. S. Patent No. 855,157, May 28, 1907.

turnings, lime and coke or coal are mixed to give a ferromolybdenum of desired proportions, and treated in the electric furnace. The furnaces are operated intermittently, *i. e.,* one charge is completely discharged before another is added. Ferromolybdenum is first made as a high-carbon alloy containing 3 to 4 percent carbon. This is then decarburized with a lime slag or a slag of lime and iron oxide. If the latter is used the percentage of iron in the alloy is increased.

Experimental Electric Smelting of Molybdenite.

An experiment was made by the writer, using the electric furnace described in the ferrochrome experiments, to test the possibility of sulphur elimination with a charge of molybdenum, excess of lime and carbon as a reducing agent. The experiment was made directly after some experiments on the production of molybdenum steel directly from hematite and molybdenite, and cannot be considered as quantitative, as the furnace lining was in poor condition. Some iron had also been left in the furnace, which diluted the product. The electric furnace was regulated so that all heating was by resistance heating only. On tapping, 1.5 pounds of ferromolybdenum were obtained. The odor of sulphur was very noticeable during the experiment. The product was very brittle, but was not extremely high in sulphur considering the amount of sulphur in the charge. There was 30 percent sulphur in the ore, and in the product 0.19 percent sulphur. A commercial 50 percent ferromolybdenum contains about 0.03 percent sulphur and 0.5 to 4 percent carbon. The coke, fluorspar and lime were of the composition given in Table I.

TABLE IV.

Production of Ferromolybdenum from Molybdenite.

Ore		Charge		Metal		Slag	
	Percent		Lbs.		Percent		Percent
Mo	47.5	Molybdenite	4.0	Mo	50.55	MoO_2	3.18
Fe	1.25	Coke	1.04	C	0.73	CaO	41.80
SiO_2	13.90	Lime	3.01	Si	0.94	MgO	12.75
P	0.608	Fluorspar	0.25	P	0.078	FeO	7.56
S	30.64			S	0.19		

From this experiment no conclusion can be drawn as to the loss of molybdenum, but in three runs made on the production of molybdenum steel from hematite and molybdenite, with carbon as a reducing agent, the average total molybdenum loss was 46.1 percent, of which 25.4 percent was by volatilization and 20.7 percent by slagging. Thus 56.2 percent of the total molybdenum loss was by volatilization.

The conclusions made from the ferromolybdenum experiments are: First, that ferromolybdenum low in carbon can be made directly from molybdenite in the electric furnace, with excess lime as a desulphurizing agent and carbon as a reducing agent; second, that a product of low percentage carbon can be made, and, third, that sulphur can be readily slagged as calcium sulphide with a charge of excess lime.

ELECTRIC SMELTING OF VANADIUM ORES AND THE PRODUCTION OF FERROVANADIUM.

Most of the ferrovanadium made is produced by the thermit process, by reduction of the oxide V_2O_5 in a crucible with carbon or by reduction of vanadate of iron with carbon in a crucible. In Europe some ferrovanadium is made by reduction of the oxide, sulphide or vanadate of iron with carbon in the electric furnace.

The largest source of vanadium is the Peru deposit of patronite, a sulphide of vanadium containing about 60 percent sulphur and 20 percent vanadium. In Colorado and Utah there are large deposits of low-grade carnotite, a uranium and vanadium oxide mineral. Another ore found there is roscoelite, which contains vanadium oxide but no uranium.

Patronite ore is first roasted and then reduced by the thermit process. Or, in the electric furnace it may be directly reduced with lime as a desulphurizing agent and carbon as a reducing agent. The other ores are treated by chemical or ore dressing processes to obtain the vanadium as oxide or vanadate of iron, and then reduced with carbon in a combustion or electric furnace or by the thermit method. With oxide the electric furnace method is similar to the production of ferrotungsten with carbon as a reducing agent. The sulphide reduction is similar to that of

molybdenite with lime and carbon. Several methods have been patented using silicon as a reducing agent in the electric furnace. The process as carried on in the electric furnace has no novel features beyond those stated regarding chromium, tungsten and molybdenum. Decarburization is with oxide of iron ore or oxide ore of vanadium. Vanadium is rather difficult to reduce, and as a result excess carbon is used, which is absorbed up to about 1 to 4 percent.

CONCLUSION.

In this paper the aim has been to present experimental data and facts concerning the electric-furnace metallurgy of some of the more rare, or at least more rarely mined, ores of the western United States. For a discussion of the general and commercial side of the electric smelting of chromium, tungsten, molybdenum and vanadium ores, reference is made to a paper by D. A. Lyon and the writer, presented before the Society in conjunction with this paper.

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A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

THE ART OF ELECTRIC ZINC SMELTING

By Woolsey McA. Johnson.

This paper has been designedly given the title "The Art of Electric Zinc Smelting." So let us inquire what is a metallurgical art. An art, generally speaking, is a skillful use of means at hand to a desired end. The subject of this discussion is, therefore, the attempt of making money out of zinc ores by electrothermic means, with emphasis on the words "making money." I suppose that, strictly speaking, there is no art of electric zinc smelting, for the commercial trials in this country, Canada and abroad have been clever, but are not art, since none of them are pronounced commercial successes.

Our ideas and practical embodiment thereof are radically different from previous commercial attempts. We are not, however, claiming anything more than our personal faith in our ability to operate our process on zinc-lead-copper ores, recovering spelter, base-lead bullion and copper matte in one operation at a probable handsome profit under certain conditions. It is evident that the only criterion of a successful new process is the actual production of finished product at a profit for the market under actual commercial conditions. With such a mental reservation on the part of my listeners and myself, *i. e.*, that we are discussing what I think may happen with the Johnson process, let us proceed to the subject directly.

The fact that a continuously operating electric zinc furnace making spelter, copper matte and base bullion, and eliminating the mineral impurities as a fusible slag, all in one operation, if made in large units would prove of decided commercial superiority to the retort plant, which is simply a laboratory experiment multiplied by 3,200, is of course as patent as the other fact that no commercial electric zinc furnace exists.

With a little personal pride we quote from a report written November 30, 1904:

"I reached Iola on July 14, 1903, and sized up the proposition as an attractive one. The weak points of the present method of treating ores seemed to be as follows:

"1. High labor charge per ton of ore treated. As labor is growing more expensive and arbitrary, and ore is growing lower in grade, it was thought that the cost of labor per pound of spelter produced would grow higher in the next ten years, as it has in the past ten years.

"2. High fuel cost per ton of ore. As fireclay is an insulator, the waste heat necessary to force the heat through the refractory material is large.

"3. Low recovery from even high-grade ores and restrictions as to nature of ores because retorts will not stand ores containing a large percentage of fluxing gangue.

"If an electric furnace could be built that would be commercial, it would, on the other hand, have the following advantages, to wit:

"1. Large units. It might be possible to make an electric retort of capacity of ten tons of spelter per day. Consequently there would be a small labor charge per ton of spelter produced.

"2. Higher thermal efficiency. For smelting zinc ores a reducing atmosphere of high temperature is needed. By the nature of electric heating a reducing atmosphere of high temperature can be maintained in an electric furnace. In electric smelting the heat is put inside the retort exactly where it is needed.

"3. It would be possible to make a durable lining to the furnace or retort because the lining can be built of special brick of such nature as to withstand corrosive charges.

"4. Great ease of control over smelting because of facilities with which the temperature of the furnace can be raised by electric heat.

"5. From reasons given above it was thought that a pure spelter could be produced from ores containing a high percentage of lead, iron and other impurities.

"The proper course was to find the right conditions on a small scale at a minimum of expense, and then to duplicate these on a larger scale."

All the above is generally much more commonly accepted as probable truth now than then by those having a knowledge of the zinc business.

Now the idea of electricity applied to zinc metallurgy is not a new one, for those brilliant idealists, E. H. and A. H. Cowles, built the first man-sized electric furnace because they owned a

zinc-copper mine in New Mexico with refractory ores. That stubborn zinc mine indeed did fertilize the American electric furnace with a true idea.

Zinc is an old friend of the goddess Electra, for the voltaic cell, with the aid of which Faraday, Daniell, Helmholtz and others did all the classical experimental work on which foundation all electric engineering is placed, was surely zinc-begotten. Without the zinc primary battery the phenomena of electrolysis and electromagnetic induction would remain today hidden in the womb of truth.

But this very friendship of electricity and zinc is seductive, for any experimenter with 20 kw. at his disposal, a few firebrick, some zinc ore and a couple of carbon electrodes cannot help making some zinc, only a few pounds, to be sure, but enough to allure him to further pursuit. And the very contrariness of the metal that seems to have so much free-will is that if one tries to convert 100 percent of zinc vapor into commercial zinc-dust, one will fail. For zinc is a peculiar metal.

All our attempts up to 1909 were concentrated on the condensation of zinc vapor to spelter. We were correct in the statement, I believe, that we knew seven or eight years ago that the main conditions for good condensation were as follows:

1. A regular flow of *pure* gas.
2. Space at right temperature.
3. Sufficient time.

As Prof. Richards says, the two latter are the fundamental conceptions of metaphysics which we learn in the first year of our life.

In the plant of the Continuous Zinc Furnace Co., at Hartford, we use the terms (1) "chemical" blue powder and (2) "physical" blue powder to denominate the two classes of blue powder. The latter is made because of bad physical conditions, and can be remelted to an ingot autogeneously by putting in a covered can and starting slow combustion by a match. The heat of the zinc-dust burning on top will melt the lower part of the zinc-dust to metal.[1]

"Chemical" blue powder is the result of poor chemical conditions or comes from the attacking of the zinc before it condenses,

[1] The theoretical conditions governing the condensation have been admirably expressed in an editorial in Metallurgical and Chemical Engineering, **10**, 451 (August, 1912).

as it condenses, and after it condenses by noxious products of smelting, making a superficial coating around each drop and so preventing the droplets coalescing. No one would, of course, ask the specific nature of these conditions, but let it be stated merely that there are a number of kinds, and that all but three of them can be easily prevented by foresight, provided their causes are known. The three kinds give positive indications of formation in our process, and the nearness of the relation between cause and effect, as shown in our smelting indications, gives us some confidence in our process. At any rate, the table given later, showing some of our last year's work, where on complex ores, high in FeO and analyzing 4 percent S, we have exceeded the condensation factor of 50 to 70 percent, obtained in retort practice on simple ores well roasted.

The new successful electric zinc smelting furnace and process will have, it is believed, the following co-ordinated parts and qualities, durable and designed with engineering skill:

1. A first-rate preheater, mechanically strong and machine operated. Preheating *per se* has no advantage save to reduce kw. hours per ton of ore. Preheating must be done right, not too hot, not too fast, not too slow, and not too cold, with a progressive application of metallurgical means.

2. The flow of heat thru the charge in the electric smelting furnace and slag must be under perfect control. The electricity must be controlled, and care must be taken that the zinc globules in eductor assume no statical charge, otherwise coalescence of the sphericles is hindered.

3. A regular flow of pure zinc gas must be delivered at proper temperature to the condenser.

4. The condenser must be durable and under good metallurgical control.

When all these conditions are fulfilled, spelter will come as a result of continuous electric zinc smelting in surprising amounts.

Something has been said about the past, and a little something about the present; now let us say a differential little something about the future.

The future of the electric zinc furnace looks big, especially on the commercial side. But the inventor has open to him further possibilities. The patent counsel of the Continuous Zinc Furnace Co. advises us to think little about the dim future, do little pro-

jective work, and concentrate upon the problems of each day. The statement of the essential advantages as outlined in 1903 and given on page 192 might be modified and the directions of future progress indicated in a general way especially as regards zinc mining.

The present Johnson zinc smelting process depends commercially upon the by-products. Furthermore, there is no doubt that our recoveries of lead, copper, gold and silver will be much higher than those of a lead furnace or of a copper furnace.

Our general proposal is to treat low-grade ores high in by-product values. While we have treated oxidized ores, as Joplin 70 percent zinc or even oxide of zinc, at the plant of the Continuous Zinc Furnace Co., we prefer to use low-grade ores analyzing about 35 percent zinc.

It seems distinctly probable that the cost of treatment of the zinc ores in the Johnson electric zinc furnace will be roughly inversely proportional to the percentage of contained zinc. Our process, therefore, will have quite an influence on the concentration of zinc ores and utilization of crypto-crystallic ores which are now commercially unavailable. It is thought by us that the general tendency, when we are successful in our commercial trial, will be to shorter and more direct concentration with as much of the Cu, Zn and Pb in one concentration as is possible. Lead smelting and copper smelting with low rate for power will be as cheap or cheaper in the Johnson furnace than in the ordinary lead or copper furnace because of the increased recoveries, provided metals stay at average prices. The relation between concentration and smelting will be different. It looks as if what the Johnson process needs were "zinc-iron middlings" with considerable included galena for maximum commercial efficiency, tho of course existing concentration methods will also be applicable. We see that the general commercial conditions governing grade of ore are diametrically opposite in the Johnson process and in the retort process, where, with the exception of dry carbonate zinc ores, the cost of treatment increases as the grade of ore treated is lowered. Metallurgical needs of the Johnson process are for fluxing ores high in lime and iron.

There was a discussion recently of the relative commercial conditions of the cost of treatment and losses as affecting concentration processes of ores suitable for the Johnson process and

the retorting process. Some curves were mentally roughly plotted, and as a result of this discussion let my belief be stated that in the first year we will smelt oxidized complex zinc ores averaging 35 percent zinc. My estimate is that in the second year we will smelt ores averaging 28 percent, and my hope is that we will finally come to a standard practice of 18 percent to 22 percent, depending upon commercial conditions.

While I hesitate to disagree with Mr. Ingalls,[2] who has studied so long on zinc problems, yet when he says that, "if there be any dreams of an electric furnace analogous to a blast furnace in which run of mine ore may be charged roughly and from which spelter, lead and perhaps also copper matte may be obtained concurrently, I fear that there will be no realization of them," I take issue direct with him. He, in my judgment, is not unqualifiedly wrong, but qualifiedly wrong. The success of our process will come at first because the Johnson furnace is similar and analogous to a lead blast furnace.

The use of ZnO prepared by a fired process from low-grade sulphide ores in an electric furnace does not appear especially attractive, for we had the idea of "igneous" concentration of zinc ores making zinc oxide on apparatus similar to Wetherill grate in 1908 with subsequent electric smelting of ZnO so produced, but put aside the idea. However, the "Truax" process for manufacture of zinc oxides out of slags has, to my notion, certain brilliant possibilities and is a triumph of American originality. It certainly looks good. The Betts process, using silicon as a reducing agent, has decided possibilities. Consideration of wet or hydrometallurgical processes for zinc ores is useless. Their metallurgy is complicated, plant expensive, and cost of treatment high and uncertain. Quantitative analysis writ large does not spell metallurgy. High recovery may some day bring them to life, but only at the time when every Italian laborer knows as much as a $1,000-a-month chemist. Their only chance, and that a remote one, is for very low-grade ores containing an insoluble gangue.

That our particular problems are those of an engineer rather than those of an inventor can be seen in the following summary of runs made in the latter half of 1912:

[2] Engineering and Mining Journal, July 6, 1912.

Table I.

Summary of Runs on Electric Furnace No. 19, June to August, 1912.

Runs 1, 4, 5, 6 and 7 on New Hampshire Ore.
Runs 1A, 2 and 3 on Roasted Joplin Ore.

	Run 1 N. H.	Run 1A J.	Run 2 J.	Run 3 J.	Run 4 N. H.	Run 5 N. H.	Run 6 N. H.	Run 7 N. H.	All
P. H. Charge smelted.....pounds..	3756	780	1310	1350	3600	1680	1224	2960	16660
Spelter made .....pounds..	602	250	403	553	691	387	244	607	3637
Accounted Zinc Factor .....percent..	60.8	123	84	97	74.3	86.5	134	**	94
Condensation Factor .....percent..	73.8	70.8	71.2	87	77.5	72.5	75.4	**	75.4
Total K-W Hours	2042	452	1007	1103	2339.5	880	671	1385	9880
K-W Hours per short ton of preheated charge.....	1090	1158	1539	1637	1298	1055	1100	938	1227
K-W Hours per pound of spelter.....	3.39	1.80	2.51	2	3.38	2.28	2.75	2.73	2.72
Thermal Efficiency .....percent (1)*	....	....	....	....	55.5	40.0	63.8	50.4	47.5
Thermal Efficiency .....percent (2)	....	....	....	....	46.3	56.8	54.5	64.0	55.3
Time of smelting .....hours..	46	11	28	27	48	19.5	9	25.5	214
Slag made (less additions).....pounds..	1000	950	280	365	434	628	1070	**	4727
Pounds of slag per short ton of charge.....	532	2440	427	541	241	746	639	**	794
Total electrode carbon consumption.....pounds..	on Runs 1, 1A, 2 and 3			38	20	8	4	11	81
Electrode carbon consumption per 1,000 K-W Hours .....pounds..	"	"	"	4.55	5.8	3.9	2.6	4.8	4.35
Average Volts	37.7	34.5	29.7	30.4	29.4	26.1	33.3	34	31.9
Average Amperes	1511	1535	1528	1736	1650	1850	1860	1962	1706
Average K. V. A.	57	53	45.6	52.8	48.5	48.2	62	67.4	54.3
Average K-W.....	58.1	43.8	52.9	40.7	60.9	45.1	74.5	54.4	53.8
Average Power Factor $\frac{\text{Av. K. W. H.}}{\text{Av. K. V. A.}}$.....	1.02	0.825	1.15	0.775	1.25	0.94	1.2	0.81	0.998

N. B.—No deduction made for periods when current was off.

* (1) Calculation based on the assumption that it required 27 kw. to keep furnace going.
(2) Assumption in this case was that a ton of charge required 600 kw. hours to smelt it.
** Mishap to furnace, not determinable.

Table II.

Summary of Runs of Electric Furnace No. 19.

Average Analysis of Slags.

	Run 1	Run 1A	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	All
SiO_2	35.96	34.98	35.77	33.56	34.48	31.52	30.57	29.20	33.25
CaO	...	6.36	8.75	12.72	8.70	...	11.86	7.60	9.33
FeO	5.16	24.55	18.36	19.40	21.50	26.20	23.80	23.38	20.42
Zn	1.54	10.96	8.26	10.51	3.7	16.5	16.2	16.4	10.52
Pb	trace	none	none	trace	none	none	none	none	...
Cu	trace	trace	none	none	none	none	none	none	...
Av. percent Zn in preheated charge	36.4	55.00	55.00	55.00	36.0	45.6	36.9	37.1	44.13

"Trace" means less than 0.01 percent.

"None" means less than 0.002 percent.

Av. sample of slags from all runs assayed 0.60 ounce silver per ton.

Table III.

Ore Used in Runs on Electric Furnaces Nos. 19 and 21.

	Joplin Ore	No. 1 N. H. Ore	No. 2 N. H. Ore
Zn	70.4	44.1	21.75
Pb	0.35	7.3	5.74
Cu	0.0	0.5	1.82
S (Total)	1.28	4.2	3.80
S (Soluble)	1.20	....	....
SiO_2	....	9.7	6.37
FeO	3.20	Fe 7.6	Fe 32.87
Al_2O_3	3.50	2.7	2.3

These ores were blended in all runs on electric furnaces Nos. 19 and 21 in several proportions and mixed with 5 percent to 15 percent flue dust and 15 percent to 20 percent reducing coal. Various fluxes were added in proper amounts.

The standard Continuous Zinc Furnace Co's mix was:

	Pounds
No. 1, N. H.	1000
No. 2, N. H.	600
Flue Dust	250

Table IV.

Summary of Runs on Electric Furnace No. 21, September to November, 1912.

All on Roasted New Hampshire Ore.

	Run 1	Run 2 (A and B)	Run 3	All
P. H. Charge smelted .....pounds..	4500	8780	7440	20720
Spelter made .....pounds..	1081	2088	1428	4597
Accounted Zinc Factor.....percent..	81.9	81.5	78.2*	80.5
Condensation Factor .....percent..	80.9	81.5	71.2*	77.8
Total K-W Hours	3932.5	6365	6426	16723.5
K-W H. per pound spelter.....	3.63	3.10	4.5	3.74
K-W H. per short ton of preheated charge	1748	1452	1728	1642
Thermal Efficiency .....percent** (1)	33.8	39.8	40.3	....
Thermal Efficiency .....percent** (2)	34.3	41.4	34.8	....
Time of smelting .....hours..	72.5	118	109	299.5
Slag made (less additions)....pounds..	302	2229	1545	4076
Pounds of slag per short ton of charge..	134.2	507	416	352.4
Total electrode carbon consumption pounds..	32	66	30	128
Total electrode carbon consumption per 1000 K-W Hours .....pounds..	5.57	8.25	3.98	5.93
Average Volts	41.6	44	41.7	42.4
Average Amperes	1761	1264	1491	1505
Average K. V. A.	73.4	55.6	62.4	63.6
Average K-W.	52.8	56	58.7	55.8
Av. Power Factor ..... $\frac{\text{Av. K-W H.}}{\text{Av. K. V.A.}}$	0.72	1.007	0.94	0.88

N. B.—No deduction made for periods when current was off.

* Split condenser pipe.

** The first calculation was made on the basis that it took 35 kw. to keep the furnace going. In the second case it was assumed that a ton of charge required 600 kw. hours to smelt it.

Table V.

Summary of Runs on Electric Furnace, No. 21.

Average Analysis of Slags.

	Run 1 Percent	Run 2 Percent	Run 3 Percent	All Percent
SiO_2	35.83	35.42	28.97	33.40
CaO	13.20	8.57	6.97	10.48
FeO	18.73	20.67	28.94	22.78
Zn	10.36	10.08	14.00	11.48
Av. percent Zn in preheated charge....	42.7	39.4	40.2	40.7

Average sample of all slags assayed 0.80 oz. silver per ton.

Trace of copper and trace of lead in slags.

$$\text{"Condensation Factor"} = \frac{\text{Weight of zinc in spelter produced.}}{\text{Wt. Zn in flue dust + Wt. Zn in ladle skimmings + Wt. Zn in blue powder + Wt. spelter}}$$

$$\text{"Accounted Zinc Factor"} = \frac{\text{Weight of zinc accounted for in all products.}}{\text{Weight Zn delivered to electric zinc furnace.}}$$

The spelter made was sold to Mr. John Conway, brass founder, 390 Broad Street, Hartford, Conn. It was used in making bronze crank cases, carburetors, steering wheels, etc., for a well-known automobile company. The castings were superior to those made when higher-priced spelter was used.

Our aim in these tests, basically considered, was to attain a condensation factor higher than retort practice. I estimate the maximum average yearly "condensation factor" attained in retort plants in the United States at 65 percent. Many retort plants in this country, however, have a condensation factor of less than 60 percent. Of course, the mixture of ores used at our Hartford experimental plant is not metallurgically or commercially possible in retorting practice.

At our Hartford plant we have mastered condensation on an experimental scale, for some of our best condensation has been with refractory ores.

It seems inevitable, therefore, that electric zinc smelting will come in due course. When we started some years ago, a little prematurely, electric zinc smelting appeared to have an indefinite future. Now the future is definitely bright.

The following paragraph from a report of November 30, 1904, is given with some pleasure:

"The big problem is the condensation of metallic vapor to fluid metal. This problem is a practical one. Electric zinc smelting requires a great deal besides a knowledge of the scientific conditions. It requires some courage to stand up, and a good deal of engineering ability to realize these conditions on a large scale. I believe that there is a good chance that it can be effected. In fact, as this world of uncertainty goes, I believe that it will be done some time within the next ten years, if not by me, by someone else. The modern industrial developments of this country, especially as affected by the labor and coal propositions, will force it on the zinc business, unless improvements in the old process are greater than I look for."

Mr. Ingalls, in his extremely clear article,[3] speaking of the claims as regards power consumption, states: "While it is doubtful whether such a result has yet been actually realized by anyone, the claim is plausible with respect to certain kinds of ores,

[3] Engineering and Mining Journal, July 6, 1912.

although even then it is necessary to assume a high furnace efficiency. It would seem that 1,200 kw. hours per 2,000 pounds of ore would be a sufficiently brilliant realization. Even such a one would put electric zinc smelting upon a sound basis in so far as power consumption is concerned." It is curious to note that 1,200 kw. hours given by Mr. Ingalls as lower probable limit is upper probable maximum limit of power consumption which I have set down for 35 percent zinc ores. Actually we have bettered this figure, and 600 kw. hours for a 35 percent zinc ore does not seem to be impossible.

With a glance at the future, and basing our reasoning on our own progress in the past three years, and extrapolating this progress, the prediction is hazarded that electric zinc-lead smelting will have a treatment cost of as low as $6.00 per ton of ore, and that the losses exclusive of roasting losses will be: Zinc 40 pounds, lead 1 pound, copper 2 pounds, silver 0.30 oz., gold 0.01 oz., per ton of ore. This is for the ultimate future. For the immediate future multiply this estimate by two and we have a safe estimate of what can be done.

It is trusted that the members of the Society will pardon the frankly expressed personal nature of this paper as well as the acknowledged tincture of egotism pervading it. It is merely the exhibition of my views on the art of electric zinc smelting as I see it in my mind's eye, and I have simply set down the thing as it is to me.

Continuous Zinc Furnace Co.,
Hartford, Conn.

DISCUSSION.

E. J. Ericson: It is interesting to note the optimism that permeates all papers on electric zinc smelting. It is well that such is the case, since a great deal of enthusiasm and courage will be needed to make it a success. By pointing out some defects I do not wish to discourage workers in this field. On the contrary, a great deal of work will be well justified, if low-grade ores not suitable for the present retort practice can be utilized.

On page 197 of Mr. Johnson's paper it is stated that the thermal efficiency is about 50 percent, but if only 10 percent thermo-mechanical efficiency was obtained in generating the electricity needed, it is apparent that the real thermal efficiency is only 5 percent, or about one-half of that obtained in the present retort practice by extraneous heating.

It is my belief that cheap electricity is the most important factor, contrary to Mr. Hansen's statement yesterday. If the electric zinc smelters in Sweden and Norway had to pay something like $26 per horsepower-year, it is probable that they would now be closed down, instead of expanding.

Mr. Johnson states further on page 200 that the "condensation factor" which in his furnace runs close to the "accounted zinc factor," *i. e.*, the extraction percentage, is only about 65 percent for retort plants. De Laval and other experimenters obtain only from 30 to 50 percent in the metallic state, the remainder being blue powder and oxide. At the Edgar Zinc Company's works an extraction from 86 to 91 percent is obtained.

In electric zinc smelting only about 80 percent total extraction is obtained, adding together spelter, blue powder and oxide. The electric furnace will have to deliver more metallic zinc and less blue powder and oxide, before it will make any serious inroads on the present retort practice.

E. F. Roeber: As the question has been raised whether these figures of Mr. Johnson for his condensation factor are not a misprint, I may say that this is not the case. Mr. Johnson claims that he has overcome condensation troubles and that he can produce at least 80 percent liquid metallic zinc.

J. W. Richards: I have seen Mr. Johnson's furnace working and I know his percentage of condensation to spelter has been as high as stated. One of the reasons why so much blue powder has been made in some of the previous furnaces like the De Laval was the overheating of the zinc vapor. If you use the zinc vapor itself as the resistor you get it superheated, and thus increase enormously the condensation difficulties, even to the extent of producing 100 percent of blue powder.

L. B. Skinner: It is very apparent that Mr. Johnson chose the title of his paper with a great deal of care. If I understand

what the word "art" means with respect to procedures of this kind, it is knowledge of the best principles and skill in the securing of a desired end.

The paper falls far short of what we might expect from its title, for the author particularly states that he has secured protection, by saying that "the patent counsel of the Continuous Zinc Furnace Company advises, etc."; we are therefore entitled to all details of the "art."

Mr. Johnson's statement of the failings of the present retort procedure is not by any means complete. In addition to the failings stated, it is clear that there are still other weak points in the present method of treating zinc ores. The practical zinc smelter might add a great many more.

On page 194, Item 1, it is stated, "A first-rate preheater, mechanically strong and machine operated. Preheating *per se* has no advantage save to reduce kilowatt-hours per ton of ore." I consider there are other desirable results obtained by preheating the ore. I think that if the inventor were operating a plant under commercial conditions, with the procedure advocated, he would find that there was one very desirable advantage obtained, and that is the thorough drying of the mixed charge. In zinc distillation, moisture has been proven to be decidedly deleterious, causing blue powder if present during the actual metal distilling period. At the beginning of retorting there is a complexity of volatile gases going off, and until certain of these gases have been driven out the condensation of metal is practically *nil,* so, owing to decomposition of the water and other physical factors introduced, moisture would be decidedly objectionable in a continuous process. Where an electric furnace runs continuously, the continuous influx of new material would add undesirable moisture, unless guarded against by the preheating mentioned.

With respect to Item 2, it has been my opinion that the crushing of ore and the drying and moving of it in devices under ordinary conditions and with consequent friction, *i. e.,* the friction of the gases against the ore particles during such operation and the friction of the particles among themselves before high temperature conditions can be reached, sets up static charges in the mass which show their greatest effect on the finer material. This,

in my opinion, causes one of the great annoyances we have in the handling of dust-forming products from feeding a roaster. After the ore in a roaster or similar device has been heated to a given stage, my observation is that "dusting" from this effect is not very noticeable. It is one of the expressions around smelters that red-hot ore does not "dust," but, after having settled these particles, I have dropped them in a proper manner from a very fine screen or some other device (so as to approximately separate them into their original sizes) without any such slow rate of settling. In fact, the settling rate of such particles would then be about a foot per second. This dust in roaster gases is analogous to what may be expected to a certain extent in handling the gases coming off from the electric furnace charge, and the remark that is made about the electrostatic charges from the zinc vapor itself might, to a certain extent, be due to the same causes or might be due to electrostatic charges on any solid dust itself affecting the condensation. Preheating might to a certain extent, therefore, cut down the production of "blue powder."

In Item 3, we have a tangible statement that it is expected to get a "pure zinc gas." I presume that by this is meant a pure zinc gas in the ordinary acceptance of the term. Of course, there are bound to be fixed gases in addition to the zinc vapor. With respect to this, I think electric zinc furnace experiments on either clean zinc ores or those containing other valuable metals have been carried on too loosely to be considered good experimental work. To get "pure zinc gas" we should be told how to avoid the production of those other volatile constituents which ordinarily occur in ores and spoil the process by forming "blue powder."

In experimenting, it should be the universal procedure to start in with pure materials, so that everything that has been put in is known, and then to make complete analyses of the products so that definite deductions may be drawn. Phenomena noted may then be properly ascribed to some one cause. Of course, everything that results from the experiment should be determined, not assumed. In other words, we should have an analysis of every product and every by-product, with definite information as to the exact conditions under which these are formed.

There would be also with the "pure zinc gas" some amount of dust, as I have stated, along with undesirable volatile matter. It may be he uses a method for purifying the gas from deleterious carbon dioxide or undesirable volatile matter after its evolution from the furnace charge. If so, we should be told the means of getting this pure gas.

In other words, we come to a matter which is one of the essential things to know in electric zinc smelting if we are to make any progress, *viz.*, what is the accurate chemical composition of the distilled matter accompanying the fixed gases? What do we form in the way of other reduction compounds, or what other elements are distilled, and what effect will they have upon the zinc vapor itself? Of the common base metals, zinc enters most readily into chemical combination, and we know very little of its chemistry beyond the soluble salts and the compounds that are used at ordinary temperatures. What will zinc unite with at an elevated temperature? It has been proved that carbon dioxide acts as an oxidizer toward zinc vapor. Are there any other elements or compounds that should be considered in discussing the actual distillation product from the furnace?

The author points out that he distinguishes between what he calls chemical blue powder and physical blue powder, but in support of that shows no analytical or physical data—only generalizations as to how he distinguishes one from the other. The chemistry not being given and the physical data not referred to in the paper, we are left a good deal at sea as to what the author actually means.

Since smelting has been confined to what is known as high-grade material, and since the object of the author's procedure is to work a complex material, we must have analyses if we are to know what effect a large lime content in the electric furnace, compared to a low lime content in the retort furnace, produces in the distillation products. We have in this new case an unknown gas and a residual mass of supposedly inert material remaining which is not comparable to the old zinc retort practice; hence assuming certain things to happen is unsafe. The addition of a large amount of lime might have an unknown effect of its own with respect to the vapors. I have seen calcium itself ignite in an electric zinc furnace experiment, and form calcium oxide.

The author says, on page 195, that he depends for commercial success upon the by-products, but we have no statement in the paper as to what the by-products are. Are they commercial? Is a base bullion produced which will be marketable? Will there be a matte of a composition which can be refined, or will we have a material which is much more objectionable than the now very objectionable lead-copper matte, which is ordinarily penalized in one way or the other for one or the other of the so-called valuable constituents. In times gone by in refining a copper-lead matte we were either penalized for the copper and paid for the lead, or paid for the copper and lost the lead. If any such penalized lead were figured in the recovery, it would be misleading. So, will the author be able to secure commercial by-products in the way of matte or base bullion?

About the volatilization of other matter along with spelter, the author expressly states that he expects, in paragraph 2, page 195, to treat low-grade ores high in by-products, and on page 196, advises that he expects, in the course of three years or so, to be treating 18 to 22 percent zinc ore. He further makes the claim that "Our process, therefore, will have quite an influence on the concentration of zinc ores and utilization of crypto-crystallic ores which are now commercially unavailable." The last part is very true. Mr. MacGregor has pointed out that mechanical separation is impossible where a very fine state of subdivision exists. In such a case, the process, if feasible as indicated, will be a God-send. But where he states, "Lead smelting and copper smelting with low rate for power will be as cheap or cheaper in the Johnson furnace than in the ordinary lead or copper furnace," I cannot for a moment understand what the author has in mind in making such a statement. It may be either due to ignorance as to present cost or recoveries in the lead smelting and copper smelting business or it must be a misprint. Taking the constituents in a charge such as he speaks of, the lead smelting part of the operation producing lead bullion or the copper smelting part allowing for the production of a matte, where the costs are now so low, I venture to say that the interest cost of the necessary equipment of the furnace itself, with amortization, etc., would approximate our present known working costs, and when it comes to a big revenue, to an increased recovery, the author would have

to make recoveries that would be so material as to run over a hundred percent in order to make any showing at all.

The author states on page 196, "Consideration of wet or hydrometallurgical processes for zinc ores is useless." With that I agree, and would like to add further as a good reason for agreeing, that usually the ore itself is put into worse shape than it existed in the mine before any work had been done on it. The heats of formation are so much higher in such processes than those of the original crude material you have to start with that final dissociation presents too many drawbacks.

Now with respect to the data offered by the author, we only get a few items out of the tables. As I have pointed out, we have no analysis of the matte and no analysis of the bullion. I presume gold and silver would be found in these complex ores, as there is in most of them, but I do not know the particular ore mentioned to be gold or silver bearing. The resumé of the runs made does not show much except the number of kilowatt hours used per ton of charge and the "condensation factor." I question the "condensation factor" of 65 percent as representing plants now operating the retort process. Mr. Ericson has also called your attention to this; you have his statement from operating plants.

To conclude, when we have an art outlined, we are entitled to all the data that can be furnished us, so as to allow of a critical discussion or an analytical study of the paper. This paper is wonderfully lacking in anything that will enable one to form any approximately correct conclusion as to what might be accomplished even when a particular ore was used. It is difficult to accept the results given, in view of the multitude of failures along similar lines, in which "blue powder" has been the main product. I have absolutely no question but that the recoveries are as stated, with respect to spelter. But the actual percentage of spelter produced is so much higher than others have ever been able to obtain, that we are entitled to know how this work was carried on to secure these results. We are entitled to know all the chemical considerations, the ideas in mind in forming the various slags and how these particular slags were formed and why others have not been tried or, if tried, were found to be failures. In other words, a general metallurgical discussion of

such matters as should have been presented when so phenomenal a recovery as 75 percent spelter was obtained. That so high a recovery was obtained should be a matter for congratulation to the author, but this amount of zinc recovered is very low in comparison with present retort practice, where one can count on from 78 to 82 percent of the zinc charged.

I am in hopes that something will come out of these electric zinc-smelting experiments and I am in hopes that the author has the right principle; but before we can accept it as such, I think that remarks such as have been made should lead to a further and much more complete exposition of the subject by the author.

G. H. Clevenger: Mr. Skinner has already touched upon one point which I had in mind. On page 195 of the paper the author makes the following statement: "Furthermore, there is no doubt that our recoveries of lead, copper, gold and silver will be much higher than those of a lead furnace or of a copper furnace." Presumably the lead and copper furnaces referred to are the standard type of lead and copper furnaces now in use. Fundamentally there would seem to be no difference between the results to be expected in an electric furnace heated by an electric current and in a furnace heated by carbonaceous fuel, as regards slag losses, etc. However, in the particular case considered by Mr. Johnson, there would be the added complication of the presence of zinc in the charge. However, we may lay this consideration aside and assume that the conditions are the same in both types of furnaces.

Most of our Western smelting plants are operated upon one basis—upon a money-making basis. In many cases higher recoveries of values (or in other words, the making of cleaner slags), would be possible if the metallurgist was not restricted to the consideration of making his plant show the maximum profit. Often it is found advisable to throw away a few cents in the slag in the form of unrecovered gold and silver and the base metals in order to save a still greater amount in operating costs.

Now, while the author's experimental results seem to indicate that lower slag losses might be attained in the electric furnace, yet I am inclined to think that when he comes to operate his furnace upon a commercial scale he will find, just as the Western

metallurgist has found, that high theoretical recoveries have to be sacrificed for tonnage and costs.

Considering the actual results in this direction claimed by Mr. Johnson on page 201, it will be found that he gives the gold value of his slag at 0.01 ounce.

F. A. LIDBURY: That is for "the ultimate future."

G. H. CLEVENGER: We will consider the ultimate future. It is true that the value of the components composing the charge have a direct influence upon slag losses, yet in a general way rough comparisons may be made with Mr. Johnson's work in the electric furnace. Taking for example the gold loss of 0.01 ounce, there is nothing extraordinary in the making of a slag containing so little gold. Those familiar with the results obtained in Western smelters know that very frequently, even when operating under the peculiar conditions which exist regarding the making of the maximum profit, slags are frequently made which do not run as high as this in gold. In fact, upon a rough estimate I would say that the average value in gold of most slags made at the present time throughout the West would not be over 20 cents per ton (0.01 ounce). The results given by Mr. Johnson for silver are most astonishing, and in fact much better than anything possible with present Western smelting practice. The loss of only 1 pound of lead in lead smelting, and of only 2 pounds of copper in copper smelting, are things which do not exist in present practice. However, no man can say just what the results would be if the metallurgist were given a free hand to obtain the highest possible recoveries regardless of the costs of operating. When viewed from the standpoint of what I might term maximum economical recovery, I quite agree with Mr. Skinner that the recoveries made by Mr. Johnson's furnace would have to be considerably over 100 percent in order to make of any importance the additional saving through the lowering of slag losses.

J. L. MALM: I would not have spoken on this paper had not Mr. Skinner referred to Mr. Johnson's statement that "the consideration of wet or hydrometallurgical processes for zinc ores is useless."

This statement is about on the order of Mr. Johnson's former discussions of hydrometallurgy. It is almost useless to reply to

an indefinite statement of this kind. There is no reason given why hydrometallurgical processes for zinc are useless. I have not followed electric zinc smelting very closely, but I recall Dr. Roeber appearing before the Teknik Club of Denver two years ago and presenting the status of electric zinc smelting On inquiry as to what had been the actual result, he stated there had been a few paper-weights made. In view of the expenditure of brains and money on electric zinc smelting and the meager results, I think it wise that metallurgists should look further and see if we have not reached a more advanced stage in other methods.

Some years ago an associate of Mr. F. T. Snyder came down from British Columbia and visited a plant which was testing out hydrometallurgical methods for zinc; he said: "I cannot see where we have a show in the matter. Your development has been a thousand percent over that reached by the electric furnace."

Mr. Skinner, I infer, thinks that hydrometallurgical processes are useless because they transform the metals during treatment into compounds that require more energy to decompose than the original combinations. The heat of formation of zinc chloride is higher than that of zinc sulphide, therefore, he argues, why should you go through the stage of producing zinc chloride from zinc sulphide in order to get metallic zinc? Such objections are unreasonable.

As cost of treatment is the determining factor, I will state that the normal zinc ores of this territory containing from 10 to 12 percent of zinc can be treated by electrohydrometallurgical methods and produce the metals in marketable metallic condition for 2½ cents per pound of metal produced.

I had hopes that at a meeting of the American Electrochemical Society in the West, electrometallurgical processes, developed and suggested, might be reviewed; there are a number advocated not only here, but in the adjoining States, and I believe a number can be brought to a successful stage if we have the assistance of the electrochemical engineers of the East. But how can we do it unless we understand the processes of electrochemistry or the engineering features involved? I know a certain proposition that has large merit and on which money is being spent, but which has no possibility of successful development in view of the fact that they are attempting to heat a solution which

contains gangue, chlorides of copper, iron and zinc, by passing steam through an immersed iron coil painted over with a coat of asphalt. You can appreciate what absolute folly it is for us to attempt the development of metallurgical processes unless we have the knowledge which will carry us beyond the use of such painted iron apparatus in contact with hot corrosive solution.

E. F. Roeber: I express the hope that the paper on the Malm process that was promised us by Mr. Malm for this meeting will be presented at the next meeting.

W. McA. Johnson (*Communicated*): I have read and re-read with interest and pleasure the criticism of my paper on "The Art of Electric Zinc Smelting." Most of it seems to be beside the mark. I ran a zinc works successfully for a period, and am familiar with present practice and present results. We hear a great deal about blue powder made by the electric furnace, but little about blue powder made by retort furnaces. I wonder if people know that 70 to 80 retorts out of 320 retorts in standard "Kansas" gas-fired retort furnaces now run on blue powder and "between products," and make per condenser 50 percent to 100 percent more metal than retorts working on "charge from mix-room." On low-grade ores, often the total metal from the "blue" is more than the metal from "charge." This is a plain fact. Mr. Ericson's criticism does not stand the test of reality, for we have exceeded the condensation factor of retorts in an electric zinc furnace.

Mr. Skinner starts in his discussion by making his own definition of the word "art." This is wonderful, because I have searched the Century, the Standard and Webster's dictionaries, and find no synonym of word "art" that resembles Mr. Skinner's definition. I had looked up the definition of "art" in dictionaries, had asked the advice of Mr. F. P. Fish, who certainly knows English and the meaning of the word "art," and had made with his help a modified definition of the word "art" in the concrete sense of "doing something." Mr. Skinner's definition of "art" as "a knowledge of how to do things" is not accepted generally. "Art" is not an abstract term, but a concrete term. Moreover, in an introductory paragraph I limited discussion by acknowledging that it was merely my own views of what might happen.

With marvelous confidence, inspired by cock-sure definition of a word to suit himself, said definition being contrary to the dictionary's, Mr. Skinner states that "I particularly call to mind that I have secured (patent) protection," etc., and that "my paper falls short of what he might expect from its title." Mr. Skinner must have forgotten that any widespread diffusion of patentable particulars would invalidate foreign patents, and also that there might be details which were not yet protected in the United States. Possibly, as he has coined his own definition of a word, he is making some new patent law for himself and imposing this on me. *Apropos* of another criticism by Mr. Skinner, I would point out to the members of the Society that on page 193 of my article I gave a specific test how to discriminate between "chemical" blue powder and "physical" blue powder by melting same under non-oxidizing conditions. It is not incumbent on me to tell Mr. Skinner all I know. I also said that "no one would, of course, ask the specific nature of these conditions." Thus it seems hardly the retort courteous, not to say the retort continuous, for a person to criticise another person for not disclosing his own knowledge, especially when it belongs to his stockholders.

Again, Mr. Skinner says that we should have treated high-grade material, etc. On page 195 of my paper it is stated that "we used roasted Joplin ores and even oxide of zinc, but prefer low-grade ores." Mr. Skinner seems to have his own slant of vision.

Now, I regard all this criticism as neither to the point nor well meant. Electric zinc smelting is much different in many ways from what outsiders imagine. A practical illustration can be given. Our slags are so liquid and so limpid that we cannot use ordinary "botting-clay" to stop the tap-hole without having an explosion. Our conditions are so radically different that few comparisons can be validly drawn by an outsider.

Mr. Skinner's point that electrostatic charges increase "dusting" is unquestionably well taken and a pertinent criticism.

Regarding his criticism as to cost of lead smelting in the Johnson furnace, I would ask Mr. Skinner to figure out a heat balance sheet for lead ore with preheating to 1050° and smelting zone at 1250° C., assuming a thermal efficiency of 25 percent

in preheating, 65 percent in electric smelting, 10 lb. (4.5 kg.) electrode carbon per 1,000 kw. hours, 11,000 B. T. U. (6,100 cal.) coal at $3.50 per long ton, power at $0.003 per kw. hour, all commercially and metallurgically possible conditions, and compare the result with the cost of smelting lead ores with coke. The figures will surprise him.

When preparing my article, I hesitated about leaving in the statement that the Johnson furnace could compete with the lead furnace, but I thought on reconsidering this question that results achieved by The Continuous Zinc Furnace Company in condensation, power consumption and electrode consumption constituted such an advance in the state of the art that the prediction by me, its president, was worthy of respect. But this claim must be left to time to prove.

This misunderstanding and misquoting and misinterpretation strike me as hardly fair. It does not induce one to contribute further to advance the state of the art. The points that should have been discussed were electrode consumption, power consumption, condensation factors and commercial developments. Were it not for the fact that art is long and life is short, I would again take up these points. But success in electric zinc smelting is not achieved by talking or writing, but rather by working and doing. We have taken our time about working out our process, but the year 1914 should show it in successful operation. This I shall endeavor to realize. At any rate, our process is founded on correct principles of chemistry and commercial science, and some time will succeed.

In fairness to Mr. Skinner and others, it might be pointed out that we ourselves have some trouble in holding in our mind proper conception of results, so revolutionary in commercial promise and so seemingly metallurgically impossible. For instance, the low values of copper and lead in slag have been checked by four technical chemists of my company, by chemists of the Massachusetts Institute of Technology, by chemists of four large industrial concerns, and by Messrs. Ledoux & Co., in order to reassure ourselves against our own doubting.

The fact that we make such good slags is explained in my discussion of Dorsey Lyon's American Institute of Mining Engi-

neers' paper. (Bulletin of Am. Inst. of Mining Engrs., Nov., 1913, page 2726-7.)

A rudimentary knowledge of physical chemistry and practical metallurgy teaches me that a smelting zone without a blast entering it and sufficiently reducing to reduce 1,000 lb. ZnO per short ton of charge would make a matte so metallic as to effectually decompose any dissolved lead sulfide. In point of fact, our mattes rarely contain over 5 percent lead, and usually contain less than 2 percent. About one-third of them analyze only a trace of lead. This is remarkable, but simple. Most of the iron oxide is reduced in the pre-heater or in the charge column to a metallic powder. This iron reacts with sulfides in the smelting zone and forms iron sulfides. It is simply, as regards smelting of lead compounds, the old "precipitation" process carried scientifically to the nth power.[1]

So, while Mr. Skinner's criticism is uttered in a spirit of skepticism and of doubt, we must make proper allowance for it. It will hardly affect electric zinc smelting, for nothing condenses zinc as well as zinc, and nothing proves as successful as success. Electric zinc smelting with proper preheating will come surely and slowly, for it is written in the book of fate. That is my vision, and there have been moments in ten years of hard work when this was so clear and so sustaining as to pay for all the work seventy times over.

[1] See: W. McA. Johnson's U. S. patent 868,345, filed Jan. 11, 1904, and issued Oct. 15, 1907.
W. McA. Johnson's Canadian patent 146,134, issued Feb. 25, 1913.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

THE ELECTRIC ZINC FURNACE

By Peter E. Peterson.

From the nature of the present retort method of recovering zinc from zinc concentrates it is almost impossible to conceive of any improvement therein that would lead to increased capacity and decreased costs, let alone the possible recovery of zinc from the abundant complex zinc ores (containing copper, gold, silver, lead and iron) at a reasonable cost and appreciable recovery of the other metals.

The metallurgy of zinc is one of distillation. The zinc must be vaporized at a high temperature and in a reducing atmosphere. Outside of the retorts the electric furnace is the only smelting device that can meet these conditions.

The electric furnace at present seems to hold out the only hope for improvement in zinc smelting.

The possible apparent advantages are: Large units, continuous feed and discharge, and the recovery of gold, silver, copper and lead in the form of copper matte or lead bullion.

The problems encountered in building a furnace to make the above advantages a reality have been many, and perhaps much time and work can be saved to others by a discussion of different types of furnaces employed.

THE FIRST FURNACE.

Fig. 1 shows a vertical section of the first furnace experimented with. This small furnace had no opening to the outside, although there was some leakage of gas around the upper electrode.

The charge consisted of a mixture of zinc sulphide, copper sulphide, lead sulphide and iron sulphide, and sufficient metallic iron to desulphurize the zinc sulphide ($ZnS + Fe = Zn + FeS$).

The furnace was connected to a small direct-current generator and run at the rate of 4 kw., the amperes varying from 40 to 50.

There was no way of telling when the charge was smelted, so a series of runs was made ranging from one to three hours. At the end of three hours the charge was completely smelted. The resulting matte showed from 1 to 2 percent Zn and small shots of metallic lead, while 95 percent of zinc in the charge was recovered in form of a high-grade spelter assaying 99.8 percent zinc.

The three-hour run was repeated, and practically the same results obtained.

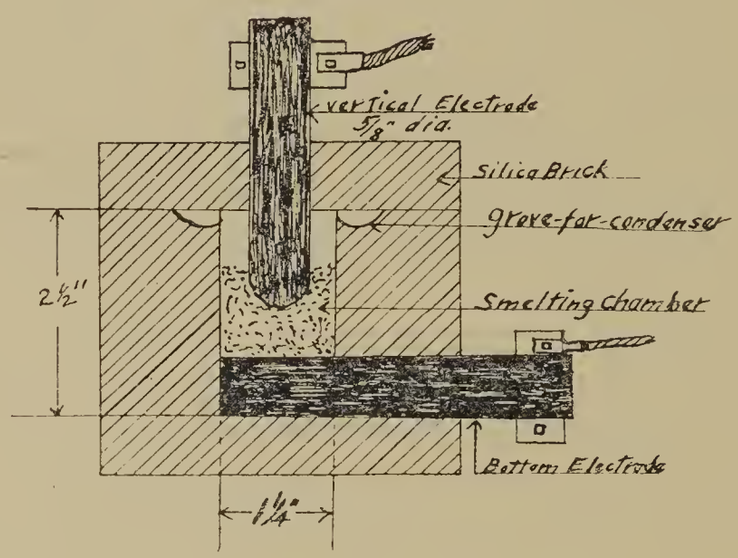

FIG. 1.

Another furnace was built with a condensing chamber on one side with a small opening for escape of gases. With this furnace it was not possible to condense the zinc to spelter, it being condensed in the form of blue powder.

LARGER FURNACE BUILT.

Results with these small furnaces were so encouraging (and there seemed to be no difficulties in condensing the zinc) that a larger furnace was built along the same lines. This furnace was constructed of ordinary fire brick, having walls a foot thick and a smelting chamber a foot square and two feet high. The top consisted of slabs of fire brick 2 inches thick, with a charge hole and an opening for the electrode. The bottom electrode was a block of iron having a cross-section of 1 inch by 12 inches extending through the wall and protruding 8 inches. The vertical electrode was a carbon rod 2 inches in diameter and 4 feet long. This furnace had a rectangular condensing chamber 6 inches by

6 inches by 12 inches. with a baffle running the long way, causing the gases to circulate first down one side and then up the other. There was a small opening ½ inch (1.3 cm.) in diameter at one side of the condenser for the escape of gases. (See Fig. 2.)

The furnace charge used consisted of unroasted zinc concentrate analyzing as follows: 47.81 percent Zn, 9.21 percent insoluble, 6.2 percent Fe, 1.5 percent Mn, 30.32 percent S, 1.4 percent Cu, 0.035 oz. Au, and 12.2 oz. Ag. Scrap iron was added to desulphurize the zinc.

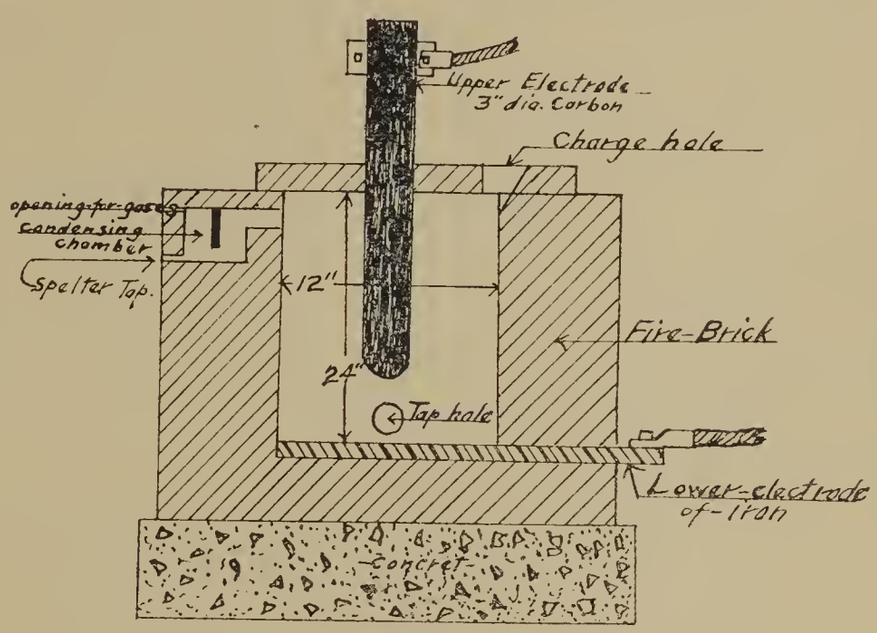

FIG. 2.

The power was obtained from a 50 kw. transformer. The electrodes were connected to a 100-volt circuit, and in series with the circuit was placed a choke coil to protect the transformers in case of short circuit and at the same time to somewhat regulate the current.

Numerous runs varying from four to twelve hours were made. The condensing arrangement failed to condense anything but blue powder. The furnace was found hard to regulate. The power consumption was irregular, and consequently the heat was the same. Attempts were made to tap the furnace, but were not very successful, although some slag and matte was tapped which analyzed as follows: Slag: Zn 0.4 percent, Cu 0.19 percent, FeO 11.1 percent, Mn 0.6 percent, SiO_2 64.65 percent, Au trace, Ag 0.35 oz. Matte: Zn 1.3 percent, Fe 60.4

percent, S 29.35 percent, Cu 1.70 percent, Au 0.03 oz., Ag 9.4 oz. The analysis of the slag and matte is interesting and showed the possibilities of the process.

CHANGE IN CONDENSERS.

The furnace was next equipped with a 2-inch (5 cm.) iron pipe 2 feet (60 cm.) long for a condenser. This pipe was flush with the inside, and stuck out a foot into the air. With this arrangement it was possible to condense some spelter after an hour's run, which allowed time to heat the condenser up to the condensing temperature. At this stage of the experimenting it was evident that for successful condensation of volatilized zinc to spelter there must be some way of controlling the temperature. The next experiment consisted of a 2-inch (5 cm.) iron pipe 6 feet (180 cm.) long extending across the top of the inside of the furnace, with series of holes bored into the upper side of pipe for the admission of zinc vapors, the theory of the apparatus being that with a long pipe, one end cold, the other hot, between these two temperature extremes there would always be a zone of the proper condensing temperature.

This condenser gave fair results. Starting the furnace cold, at the end of an hour it was noticed that condensing commenced, and would continue at a good efficiency for an hour, when the condenser seemed to get too hot, or, rather, the zone of proper condenseing temperature was greatly reduced in area. Various means were tried to increase the length of this condensing zone.

A large pipe 8 inches (20 cm.) in diameter was inclosed with brick and heated with coal. Clay tubes of varying lengths and thickness were tried. Iron pipe insulated with varying thickness of asbestos and chambers of varying dimensions in the wall of the furnace were experimented with, but none of these gave better results than the 2-inch pipe 6 feet long.

The problem seemed to resolve itself into three parts, that is, a certain temperature was found to be necessary as well as a certain area, and that this area must not be too far away from the source of distillation. In other words, the zinc must not be kept in vapor form too long before condensing. The furnace seemed to stand up well, except the cover, which usually did not last more than a couple of days.

FURTHER DEVELOPMENT.

The bottom electrode, although of iron, gave no trouble, which was, no doubt, due to the shortness of the runs. Up to this time there was no very definite idea of costs, such as cost of electrode and power consumption. Another furnace was therefore built of the same type, and an effort made to run a ton of zinc concentrates in one run, keeping careful record of the power and electrode consumption, disregarding entirely the condensing of the zinc to spelter. This run was in two parts, one using metallic iron as a reducing agent, and the other using lime and coke according to the following reaction:

$$ZnS + CaO + C = Zn + CaS + CO.$$

The following results were obtained:

	Reducing Agent	
	Iron	Lime and Coke
Kw. H. consumed	360	388
Pounds ore smelted	455	363
Lbs. reducing agent	193	Lime 164 Coke 33
Total lbs. charge	648	560
Kw. H. per lb. charge	0.556	0.692
Kw. H. per lb. zinc concentrate	0.79	1.07
Kw. H. per ton zinc concentrate	1580	2140
Percent of zinc extracted (obtained by difference)	90	50

The above test, using iron as a reducing agent, was made in three separate runs, the furnace being allowed to cool down between runs. In using lime and coke the furnace was allowed to cool down four times. Copper-coated carbon electrodes 3 inches (7.5 cm.) in diameter were used, and the carbon consumption per ton of zinc concentrate was 12 pounds (5.45 kg.). This furnace was lined with magnesite brick. The run did not show them to be superior to fire brick. Under these conditions the cover of the furnace suffered the most and had to be replaced twice.

The power consumption varied greatly, and it required constant moving of the electrode by the operator to keep the furnace running. The furnace was charged once an hour by the means of a hopper with a slide in the bottom. Immediately after charging, the zinc vapor issued from the ends of the condenser with great velocity, due to the small amounts of moisture present. The

charge was not preheated as it should have been. There was no separation of slag and matte. An analysis of the material tapped from the part of the run using lime and coke for reducing agent is as follows: Insoluble 8.6 percent, FeO 13.2 percent, S 24.3 percent, Zn 24.3 percent, CaO 25.1 percent.

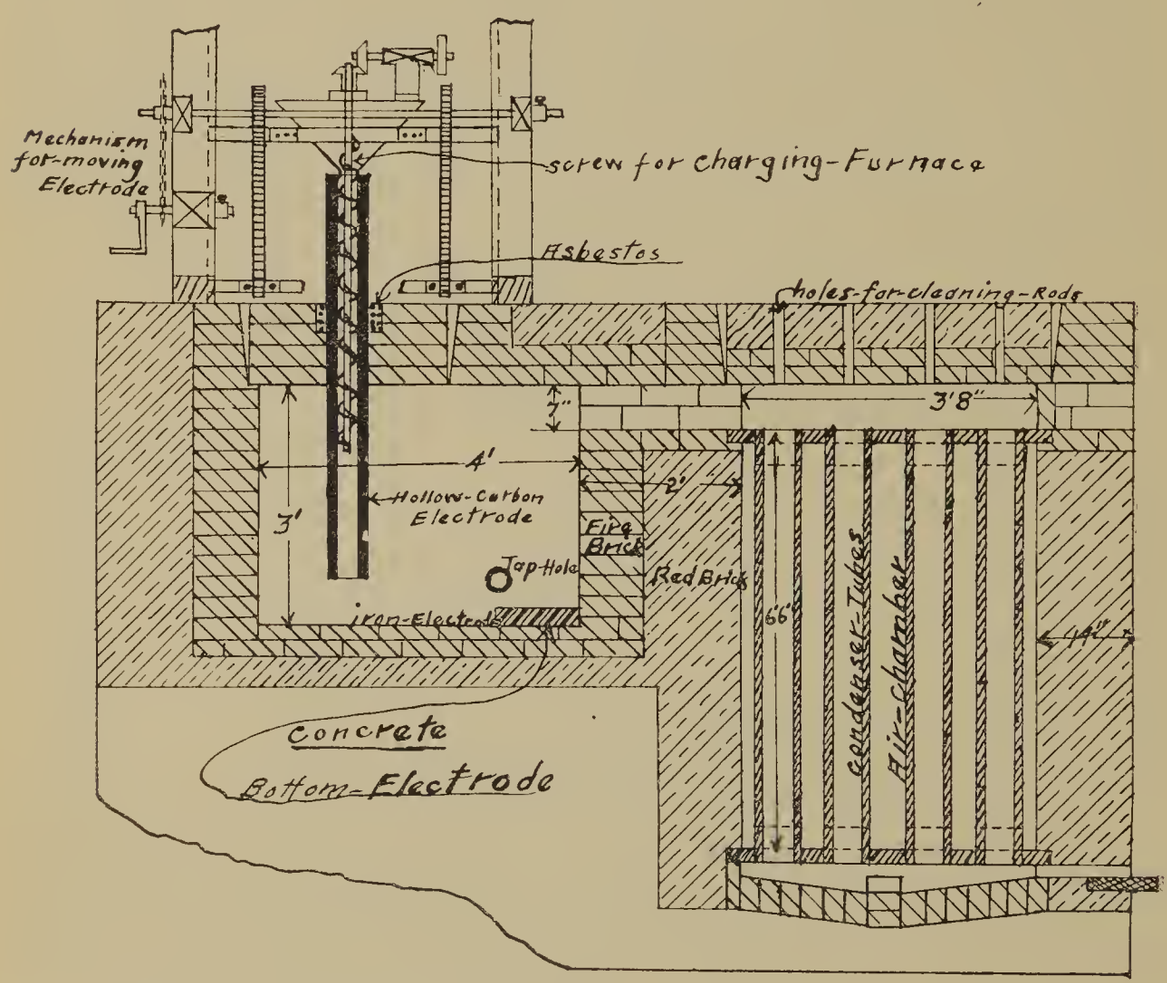

FIG. 3.

At this time it was decided that the most economical method of treating zinc concentrates could be attained by roasting them to oxide but leaving sufficient sulphur to form a matte as a collector for the precious metals. After some experimenting the practicability of the following reaction was proved:

$$Fe_2O_3 + 3C + 2ZnS = 2Zn + 2FeS + 3CO.$$

By this method the major part of the zinc is reduced from the zinc oxide by carbon, and the zinc in the sulphide form is reduced by iron which has been reduced by carbon from iron oxide.

THIRD FURNACE BUILT.

The next furnace constructed was to have a capacity of one ton of roasted zinc concentrates per day. The furnace was a radical departure from anything previously tried. The charge was fed into the furnace through the upper electrode so as to introduce it directly into the arc. There were eight clay condensing tubes inclosed by heavy brick walls, with an arrangement to cool the tubes when they rose above the condensing temperature. (Fig. 3.)

The condensing tubes being inclosed in heavy brick walls, it was thought that in time, about two or three days, the condensers would become overheated from the zinc vapors, and then it was proposed that the tubes be cooled to the right condensing temperature by air. This condensing temperature we were led to believe was between 450° and 515° Centigrade. Ingall's book on zinc smelting gives these figures. Later this condensing temperature was determined to be above 864° Centigrade. This is also consistent with theory, when it is considered that zinc boils under normal conditions at about 920° Centigrade.

CONDENSER DIFFICULTY.

After several runs lasting from two to six days it was found impossible to heat the condensers to 450° Centigrade, let alone 864° Centigrade. The zinc was condensed in the form of blue powder, as was to be expected.

The brick work in this furnace was put in with all possible care, yet frequent explosions resulted from the leakage of CO gases into the air chamber surrounding the condensers. The openings were continually choking up with blue powder, and it was found rather hard to keep them clean. This character of condensing apparatus was too complicated to offer any success.

The means by which improvements in smelting were expected gave considerable trouble. The furnace was connected up without any regulating device to control the power, and as a consequence it was found impossible to keep the temperature constant. The charge was intended to be fed continuously through the hollow electrode by a screw, but, due to variance of fusing rate, the feed was continuously choking. The object of charging in this manner was to obtain lower power consumption per ton of charge. This was accomplished.

MECHANICAL DIFFICULTIES.

The power consumed was from 1,100 to 1,300 kw. hours per ton of 50 percent zinc concentrate, but the mechanical difficulties still existed.

The hollow carbons gave considerable trouble by breaking. Later graphite tubes were used, which gave better results. The bottom electrode, which was of iron, as in the former furnaces, melted, and destroyed the bottom of the furnace with it. The furnace cover showed no deterioration whatever.

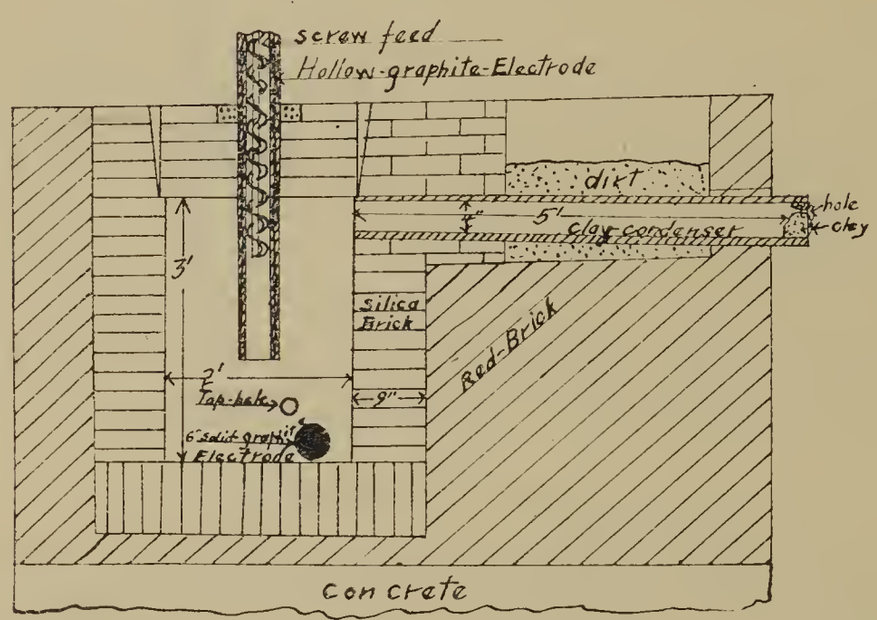

Fig. 4.

The furnace was then remodeled. (See Fig. 4.) In place of the iron electrode in the botton a 6-inch (15 cm.) diameter graphite electrode was substituted. The smelting chamber was reduced in size, and a water rheostat was placed in series with the current as a means of controlling the power.

The condensing apparatus was replaced by three clay tubes 1 inch (2.5 cm.) thick, 4 inches (10 cm.) inside diameter and 5 feet (150 cm.) long. These were placed in a shallow brick chamber open at the top. These condensers were covered with varying thicknesses of dirt, as it was thought necessary to maintain the condensing temperature. With this device some spelter was obtained. From 4 to 5 inches of each tube was doing the condensing, and the greater part of each tube was too cold to condense. The condensing apparatus was next replaced by eight

tubes 1 inch (2.5 cm.) thick and 4 inches (10 cm.) inside diameter, all of different lengths, varying from 14 inches (35 cm.) to 28 inches (70 cm.). Two of these tubes were of carbon, the rest were of clay, and two of the clay tubes were partially filled with charcoal.

The ends of these condensers were luted with clay, leaving about ⅝ inch (1.5 cm.) diameter hole for the escape of CO gases and uncondensed zinc vapor. Pyrometers were constantly kept in the tubes. No zinc could be condensed unless the inside surface of the condensers was above 840° Centigrade, and at 900° Centigrade the condensers were too hot. The carbon tubes condensed no better than the clay tubes; the clay tubes containing charcoal did not condense quite as well as the tubes without charcoal.

At no time during this run was the zinc completely condensed, for there was always uncondensed zinc burning at the ends of tubes, and at all times the condensers would condense spelter if the temperature was around 864° Centigrade. From these results it was concluded that each of the tubes had a limited condensing capacity, and if the tubes were kept at 864° Centigrade, and the amount of zinc volatilized kept within the limits of the condensing capacity of tubes, there would be practically a complete condensation of the zinc.

PRODUCTION OF SPELTER.

Another change was made in the furnace. Solid graphite electrodes 6 inches (15 cm.) in diameter were used in place of the hollow ones, and the charge was introduced by means of a screw through one side and near the top of the smelting chamber. The furnace was run on barren charge until the condensers attained a temperature of 859° Centigrade; then a charge containing zinc was introduced and fed at a rate so that very little zinc appeared in the flames burning at the end of the condensers. This was maintained for twelve hours, and the spelter condensesd and recovered was 86.4 percent of the amount charged into the furnace. Thus at last it was proved that spelter could be made with the electric furnace, but it was observed that the power consumption was approximately 4,000 kw. hours per ton of 50 percent zinc concentrate.

The furnace bottom was giving trouble. It melted out almost as readily as the charge. This complicated matters, making the slag thick and pasty, and only occasionally did any matte appear with the slag, but the matte was always found when a furnace was torn down. The bottom electrode was consumed at about the same rate as the vertical one. This and other reasons made the furnace exceedingly hard to control. The destruction of the furnace bottom was attributed to the arrangement of the electrodes.

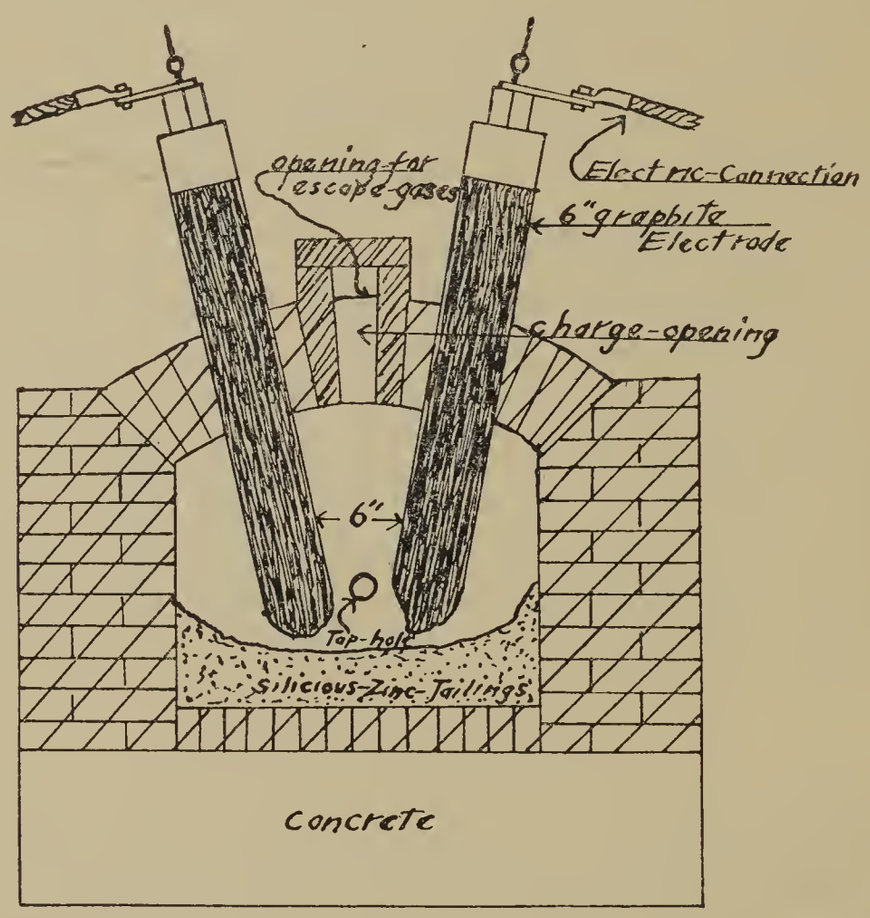

FIG. 5.

The condensation of zinc requires constant conditions, such as a quite uniform flow of gases and a constant temperature which can be easily varied. The next step was to improve the smelting part.

A small furnace was erected with two electrodes through the top. (See Fig. 5.) These were placed about 6 inches (15 cm.) apart. The inside of the furnace was lined with silica brick, and the bottom covered 10 inches (25 cm.) deep with silicious zinc tailings from mill jigs.

No efforts were made at the condensation of spelter, the zinc fumes passing into a flue and allowed to go to waste.

With this furnace numerous runs were made covering a period of thirty days. Slags of different composition were tried and the proportion of reducing carbon was determined. This work resulted in much useful information concerning furnace charges. We had no difficulty whatever in making slags and mattes and tapping them from the furnace, and the power and temperature could be kept constant or varied at will with practically no attention. Test runs on recovery of metals and power and electrode consumption were very satisfactory. The furnace was torn down, and showed practically no deterioration whatever; in fact, part of the tailings in the furnace bottom were not altered.

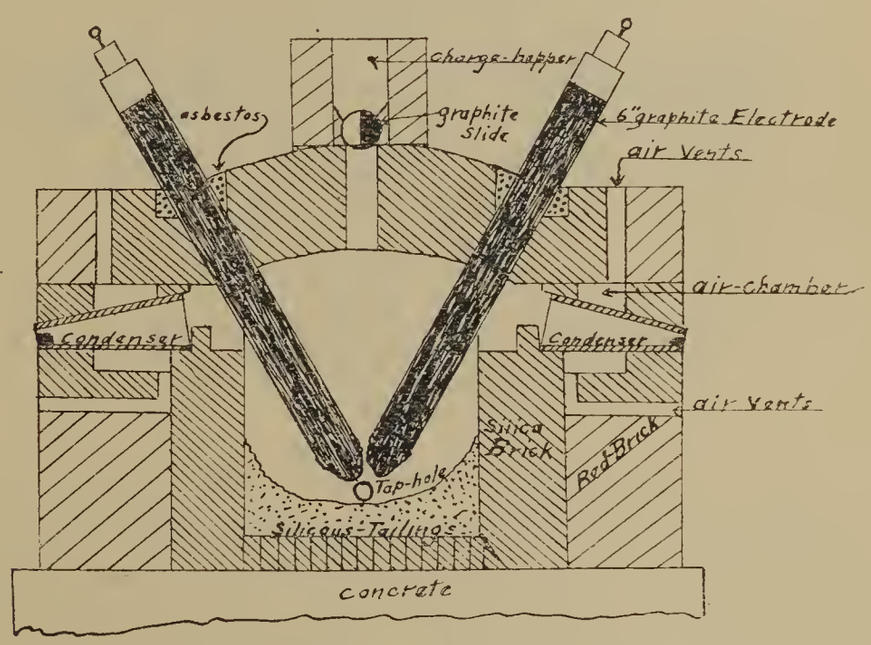

Fig. 6.

CONICAL CONDENSERS.

A larger furnace was constructed along these lines, fitted with eight conical condensers such as used by the zinc smelters in Kansas and Oklahoma.

A run was started, and for the first twenty-four hours was encouraging. Then the old trouble returned. The furnace would not respond to regulation, and no slag could be tapped. The apparent electrode consumption was enormous; 26 feet (780 cm.) of 6-inch (15 cm.) diameter solid graphite electrodes were fed into the furnace in six days' time.

Something was radically wrong, and on opening the furnace it was found that a hole six feet (180 cm.) below the bottom had been melted out. The electrode consumption had been very little; the electrodes when removed were 12 feet (360 cm.) and 14 feet (420 cm.) long, respectively. The trouble was caused by the arrangement of the electrodes. The operators in attempting to regulate the power had lowered the electrodes through the bottom of the furnace.

The hole in the bottom of the furnace was filled with silicious zinc tailings. and the electrodes placed at such an angle to each other (see Fig. 6) that when lowered they would meet at the bottom of the smelting chamber. The screw feed was aban-

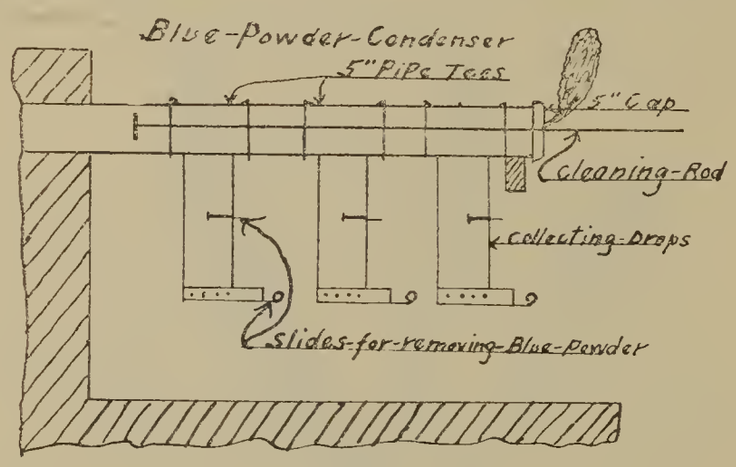

FIG. 7.

doned, owing to the irregularity of air supply used in running it, and charging of the furnace was accomplished by means of a brick hopper with a graphite electrode for a slide in the bottom.

The furnace as now arranged was smelting for an actual running time of thirty days with five stops, during which time the furnace was allowed to get cold. During these runs the previous condensing experiences were repeated without any improvement. The spelter that was condensed was found remarkably pure, assaying from 99.3 to 99.8 percent zinc. The furnace gave no trouble in any way. Slag and matte were tapped every twelve hours.

For the time being the efforts at improving the condensers were abandoned and arrangements were made to recover all zinc products. To accomplish this a new furnace was built and special iron condensers were made to recover the blue powder; also a bag house was used to catch the zinc oxide in

fumes. Fig. 7 is a view of the blue powder condenser. A twelve-day run was made without difficulties of any kind. The charge was fed in every hour, and slag and matte tapped every twelve hours.

The metallurgical results will follow.

This furnace in its present improved form is about all that can be expected of any furnace. There are far less difficulties encountered than in running an ordinary blast furnace. The deterioration of the furnace is small. Such a furnace should run from six months to a year without any extensive repairs.

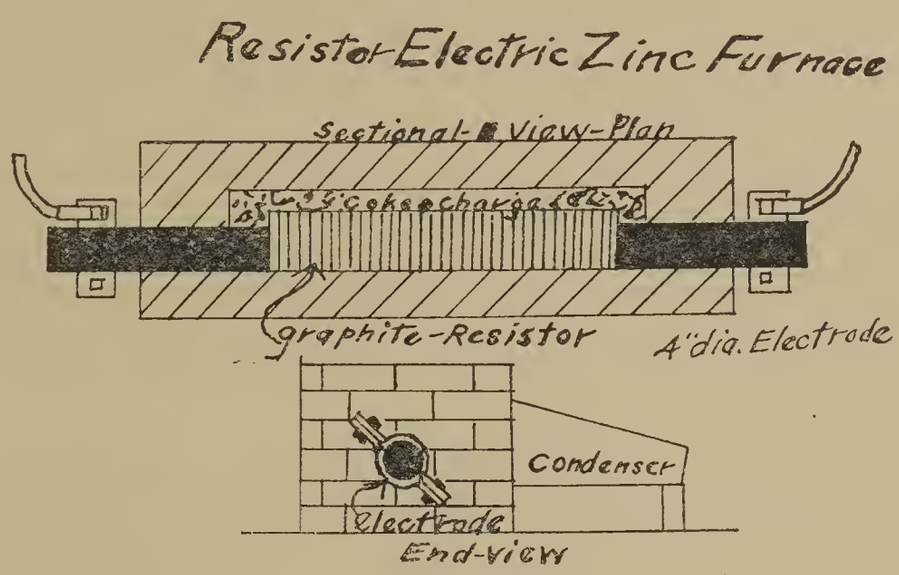

Fig. 8.

Large units can be constructed by merely the addition of more electrodes, and there is no reason why units of 25 to 50 tons each could not be operated as easily and cheaply as the one-ton units.

In connection with the condensing experiments a small resistor type of furnace was constructed. (See Fig. 8.) This furnace was made with a view to duplicating the conditions existing in the present-day zinc retort, with the exception that electrical heat was used instead of gas or coal. The essential parts of the furnace were a rectangular brick chamber having a resistor of graphite blocks in the bottom, connected to an electrode at each end. The arrangement of this furnace was such that the heat was always under control. There was one condenser of the same type as used in retort smelting. The furnace was charged intermittently with roasted zinc concentrates and coke and coal.

In operating this furnace great care was taken in regulation of the heat so as to have the same conditions as in the retorts. It was noticed that the zinc began to volatilize before the condensers were hot enough to condense it to spelter, and that after the condensers had reached the right temperature there was more zinc volatilized than it would condense; and when the heat was reduced to cut down the volume of zinc vapor, the condenser would get too cold. This condenser when at the right temperature, which could be easily maintained, would condense two pounds of spelter per hour with approximately the same amount of zinc vapor passing through without condensing. There could be no doubt whatever as to the gases being of the same composition as in the retort.

These experiments were repeated a number of times, and the results were always the same, and to me they prove conclusively that the failure to condense zinc to spelter in the electric furnace is due to the difficulties in maintaining sufficient condensing area at the right temperature near the source of volatilization.

METALLURGY.

In the operation of the electric zinc furnace the composition and condition of the smelting charge is quite as important as the furnace. More care must be exercised in making up furnace charges than in ordinary blast furnace practice. The charge must be such that at all times there is a perfect reducing atmosphere in the furnace; the slag must be such that deterioration of the furnace lining is reduced to a minimum, and with a sufficiently high formation temperature to volatilize the zinc. A matte must be made that is uniform in composition and is readily tapped from the furnace, and made in sufficient quantity to effect a good saving of copper, gold and silver. The proportion and nature of the reducing carbon is important. As before mentioned. a perfect reducing atmosphere must be maintained, and at the same time the reducing carbon must be completely consumed. If an excess is added, it can not be burned by an air blast as in blast furnace practice, and the only oxygen available for its consumption is that held in combination by the constituents of the furnace charge.

The physical character of the charge is quite as important as

the chemical for the best work. The charge constituents should be finely divided so as to allow the various reacting elements to be in intimate contact with one another, thus insuring more perfect and economical results. But the charge must not be too fine when charging in loose rather than in brickette form. The particles should be of such weight or size as to readily settle from the zinc vapors and gases passing outward into the condensers. If the particles are too fine they are apt to find their way into the condensers in the unmelted condition.

It is necessary to have the charge absolutely dry. The presence of water even in small quantities is serious, due to the oxidizing of the zinc vapor by decomposition of the water. What is still more serious is the instantaneous generation of steam and other gases, causing a great increase in the velocity of gases, thus disturbing the condensation of the zinc in the condensers and thereby causing a loss of zinc and also a loss of silver and other metals, due to furnace charge being blown outward into the condensers.

For reasons above outlined, if it is not possible to mix the charge with hot calcines from the roasting furnace and then immediately feed it to the electric furnace, there must be a preheating furnace to heat the charge before introducing it into the electric zinc furnace.

SLAGS.

The making of slags is governed by the nature of the furnace linings, and these are dependent on the nature of zinc concentrate or ore to be smelted. If the concentrates are high in iron and low in silica, then a basic slag high in iron is made, and consequently a basic furnace lining (such as magnesite brick) is used; and if the reverse is the case, then high-silica slags with silicious furnace linings are used.

The principle used in the elimination of zinc from slags was to make slags of such high formation temperature as to practically volatilize most of the zinc before slagging of the charge begins.

In silicious slags the high percentage of silica is the principal controlling factor. In basic slags the percentage of reducing carbon is increased, which for practical purposes gives the same effect.

In the work I have been identified with we have had practically no difficulty in eliminating zinc from either basic or silicious slags. The degree of elimination depended on the quantity of power used. Following are analyzes of slags showing how zinc elimination can be varied:

	Zinc	Copper	Iron	Insoluble	Silver	Gold
Slag No. 1.....	0.3%	0.24%	16.1%	66.2%	1.2 oz.	trace
Slag No. 2.....	1.6	0.13	19.2	57.5	0.6	trace
Slag No. 3.....	5.5	0.47	22.9	45.5	4.1	0.01 oz.
Slag No. 4.....	8.5	0.38	23.5	44.4	2.1	trace

MATTE.

In the treatment of complex ores the other metals, gold, silver and copper, are also recovered. This is accomplished by producing a collector (matte) similar to the lead button in crucible fire assaying.

In the earlier work it was attempted to use as a collector a matte consisting principally of iron sulphide, but its use was not attended by any degree of success. The matte formed was very irregular in composition, varying from metallic iron to iron sulphide. The amount of matte formed was also irregular, and it was found necessary to add copper ores until the furnace charge would assay in the neighborhood of 1 percent copper in order to get a matte uniform in quantity and composition. The quantity of matte was varied by varying the percentage of sulphur in roasted zinc calcines. Zinc calcines were successfully treated that varied from 2 to 15 percent sulphur. The best results were obtained between 5 and 7 percent sulphur. One of the important reactions in this connection is:

$$Fe_2O_3 + 3C + 2ZnS = 2FeS + 2Zn + 3CO$$

or $Fe_2O_3 + 3C = 2Fe + 3CO$ and $2Fe + 2ZnS = 2Zn + 2FeS$.

REDUCING CARBON.

Coke was found to give the best results. When coals were used it was noticed that the condensing of the zinc was seriously interfered with, due to deposition of fine carbon or soot in the condensers from the decomposition of the hydrocarbons in the coal.

As a basis of determining the amount of reducing material, there was computed from analysis the percentage of oxygen present in a unit charge, and coke was added in sufficient quantities to form CO with the total oxygen. Usually a trial run was necessary to determine the right proportion. The evils of an excess of coke are the formation of infusible compounds and the consequent difficulties in tapping the furnace.

The immediate remedy is to add charge without carbon until the excess of coke is consumed. This is readily determined by the physical character of the slag.

RECOVERY OF LEAD.

The lead in the ores experimented on was low, varying around 1 and 2 percent, and in all cases it was almost completely volatilized with the zinc. But I do not see any reason why zinc-lead concentrates could not be successfully treated. It would require a varying of the heat so as to reduce lead first, which, if necessary, could be tapped from the furnace, and then continue for the volatilization of the zinc—all of which is well within the limits of the electric zinc furnace.

TREATMENT OF BLUE POWDER.

At the present time the only drawback to electric zinc smelting is the inability to condense completely the zinc volatilized to spelter. Of course, this refers to operations conducted on a commercial basis, and not to small test runs where care and attention can be exercised, which is impossible on a commercial scale.

The zinc vapor as now condensed in the electric zinc furnace is mostly metallic zinc, but the greater part is so finely divided that it is impossible to melt into bars. This is called blue powder or zinc dust, the name blue powder being derived from its physical appearance. Blue powder, as a rule, has the same chemical analysis as spelter; the difference between them is purely physical. Attempts to melt blue powder in open vessels have up to this time proven failures, due to the oxidation of the minute particles of zinc to zinc oxide before consolidation of the particles could take place.

Blue powder can be melted into spelter by the use of pressure

and heat. In the experiments performed by the author (which were not original) this was accomplished by placing the blue powder in a cylinder having notches at one end. This cylinder was placed on a solid block in an upright position, with the notched end down so as to allow for the escape of the molten zinc. Into the other end of the cylinder was placed a loosely-fitting plunger connected to a screw so that pressure could be applied. Into this cylinder blue powder was placed and put under pressure by means of screw and plunger. The cylinder was then heated up to temperature from 600° to 700° C. By this means from 80 to 85 percent of the zinc could be squeezed from the blue powder.

Another method was tried—a hot electrolytic process devised by Warren F. Bleeker and described by him in the Transactions of the American Electrochemical Society, **21**, 359 (1912). In this process a molten bath of zinc chloride is made use of. The zinc chloride is placed in a vessel which is a good electrical conductor, such as iron or graphite. This pot is connected electrically as the cathode of a direct current circuit, and into it is placed a graphite or carbon electrode which is connected as the anode in the same circuit. The bath is then maintained at a temperature above that of molten zinc, and direct current passed through it. The blue powder to be melted is stirred into the bath, where it very quickly melts into spelter and settles to the bottom of the pot, whence it can be removed by tapping.

Experimenting with this process was not extensive enough to warrant giving any figures. It might be noted, however, that, with the bath in good fluid condition, and with all electrical connections clean, as much as 10 pounds (4.5 kg.) of spelter was melted from blue powder with an expenditure of only one kilowatt hour of power.

The experiments were conducted in an iron pot, and, further, the blue powder used in tests was condensed in iron condensers. This made the resulting spelter high in iron (from 1 to 2 percent). Efforts were made to refine this spelter by the ordinary fire methods, that is, by keeping the impure zinc in a quiet state of fusion near its melting point, by which means the impurities, iron and lead, would settle to the bottom, the lead almost in the pure form, and the iron as zinc-iron alloy analyzing as follows:

Zinc 91.3 percent, iron 5 percent, and lead 1.5 percent. This method of refining gave, roughly, 50 percent of the spelter suitable for market, and over 90 percent of the lead could be recovered, but the zinc-iron alloy, representing about 50 percent of the zinc recovered from blue powder, could only be placed on the market by selling to retort smelters, where it would be redistilled.

Some experiments were conducted with a view of devising a process of refining this zinc-iron alloy without distillation. Iron and sulphur unite readily at the melting point of the zinc-iron alloy to form iron sulphide. A scheme of removing the iron from this alloy by means of sulphur looked very promising, but, as sulphur vaporizes at a temperature below that of melting zinc, some other means had to be used in introducing the sulphur. Iron pyrite (FeS_2) breaks down at 565° C., giving up half of its sulphur.

Several lots of zinc-iron alloy were melted, and into them was stirred finely ground pyrite (FeS_2), while one experiment was tried with galena (PbS). The results showed that in every case the iron content was reduced (the liberated sulphur uniting with iron forming FeS, which is infusible at temperature of test), and, while the experiments were not complete, the results were encouraging and indicate that this class of impure spelter may be refined by other and cheaper means than by distillation.

DESIGN OF A MORE EFFICIENT CONDENSER.

It has been demonstrated that zinc vapor can be condensed to spelter with a fairly good efficiency with the electric zinc furnace for short periods where great care is taken to maintain the right temperatures. In the present retort method of distilling zinc it has been shown that about 7 percent of the heat in the coal is actually utilized for the work of vaporizing the zinc, and 93 percent is lost by radiation, etc. A great part of this so-called lost heat is used in heating the enormous mass of brick in the furnace, and this in turn tends to keep a certain constant temperature around the furnaces and condensers, and without this heat (or, rather, if 93 percent of the heat was utilized for the vaporization of the zinc, and only 7 percent lost by radiation,

etc., which is nearly the case with the electric zinc furnaces), I am satisfied that the retorts would not produce a greater percentage of spelter than the electric zinc furnace.

Electric zinc furnaces constructed on the same principles of heating as retort zinc furnaces have given as good condensing results. In these furnaces the heating of the furnace charge is accomplished by the radiation of heat from an electric arc located above the charge. Practice has proven this a very inefficient means of electric heat, yet so far as condensing is concerned it gives the best results.

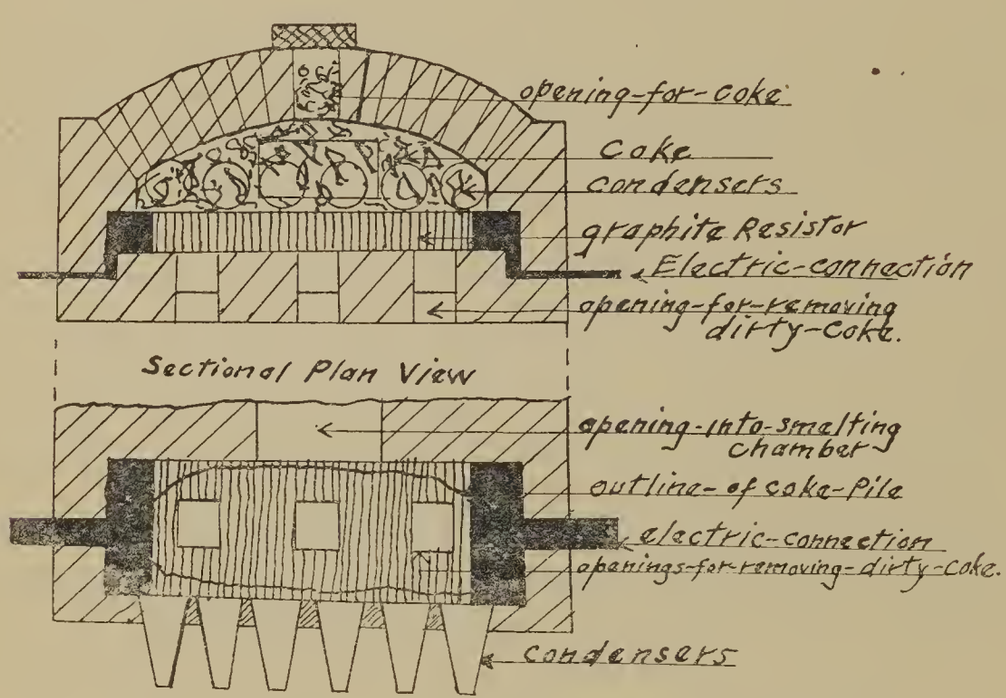

Fig. 9.

This design of an effective condenser seems to involve either the duplications of retort conditions, as in Fig. 9, or something radically different, as in Fig. 10.

The condenser as shown in Fig. 9 is so constructed as to duplicate the conditions in retort zinc distillation. It consists of a separate condensing chamber, the floor of which is a graphite resistor for electrical heating, by which means a pile of coke resting on this resistor is maintained at any desired temperature necessary for condensing zinc vapor to spelter. The coke acts as a filter, a reducing agent and a baffle. The condensing chamber is made of such a size as to reduce the velocity of gases

to such a degree that most of the zinc will have time to condense in passage through the condensers. One of the important features of this coke pile is the removal of mechanically carried dust from the distilled vapors. This dust, as a rule, is rich in silver. The coke as it becomes dirty can be removed and used as a part of the reducing carbon for the furnace charge.

This type of condenser would be large and cumbersome. One clay condenser would be required for about 45 pounds of spelter condensed per twenty-four hours. The condensing apparatus would be much larger than the electric furnace. The heat necessary to maintain right condensing temperature would increase the power consumption per ton of charge smelted.

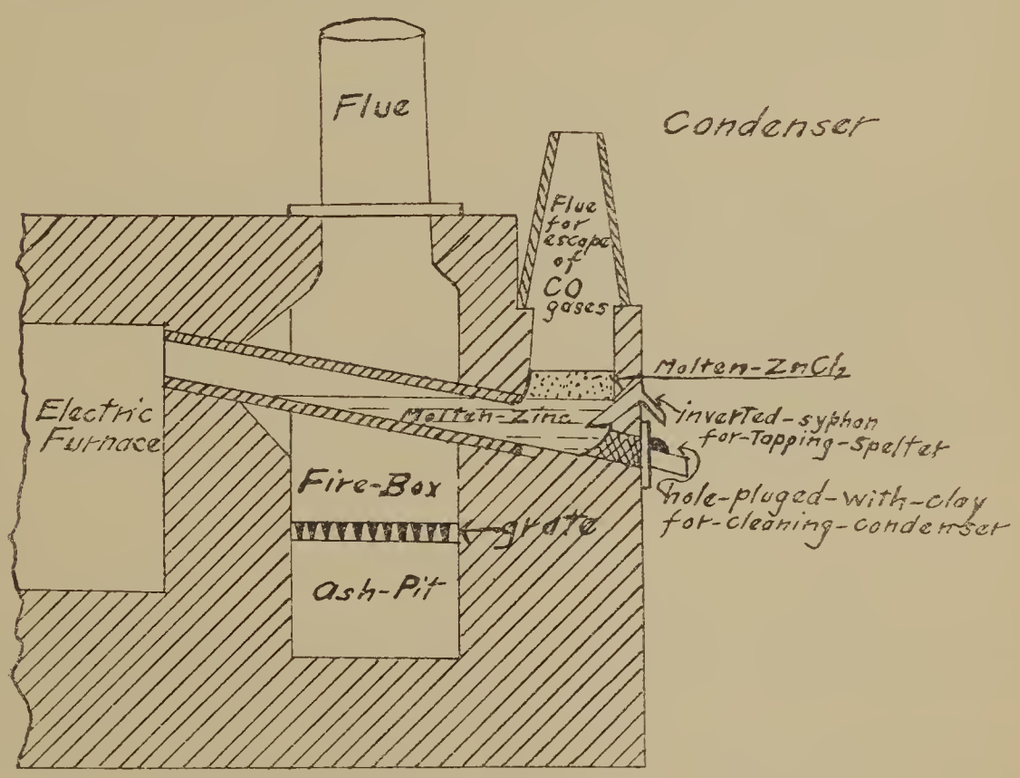

FIG. 10.

Fig. 10 shows a proposed condenser for electric furnaces that should have a large capacity and be effective in its operation. The principle of the condensing is to pass the gases from the electric furnace through a molten bath of zinc; the bath is automatically maintained at the right condensing temperature by being covered with a molten bath of zinc chloride which boils at a temperature which will not allow the molten zinc bath to get too hot. The passage leading to the molten zinc is heated

to prevent any condensing and consequent clogging of the tubes.

For the operation of this type of condenser the zinc vapors must be distilled under pressure; for observations made with this idea in mind there should not be any difficulty in maintaining a pressure of 2 pounds per square inch (0.13 kg. per sq. cm.), which should be sufficient to work this condenser.

The quickest and most effective method of condensing any vapor to liquid is the one mentioned above. A good illustration of the method of condensing is the passing of steam into water.

APPLICATION OF THE ELECTRIC ZINC FURNACE.

The following, worked out on a one-ton unit, will give a fair idea of the costs:

Costs Per Ton of Pay Charge.

Roasting	$ 1.00
Supt. and Assay Office, etc.	1.00
Labor based on 33 ton plant	.83
Power—1401 Kw. hours, @ 0.332 cent	4.65
Depreciation of plant	.40
9.5 lbs. electrodes, @ 14 cents	1.33
0.1215 tons coke screenings, @ $5.00 per ton	.60
440 lbs. matte. Cost of marketing	2.86
Total cost	$12.67

With the electric zinc furnace the following extractions can be depended upon for the above costs: Zinc extraction, 85 percent; copper extraction with 5 percent copper in charge is over 95 percent, and with 1.5 percent copper in charge is over 84 percent; silver extraction, 65 to 72 percent; gold extraction, from 98 to 100 percent.

The gold, silver and copper are recovered in the form of a copper matte. The difference in the value of the metals and that actually obtained from them is called the cost of marketing the matte.

The zinc as obtained with the process at its present perfection of condensation with the above costs contains only 30 percent spelter ready for the market. The remaining 70 percent of the zinc is obtained in the form of an 80 percent zinc product,

as blue powder and some zinc oxide. This zinc is produced by the electric zinc furnace would be worth $4.36 a hundred pounds when spelter is selling at $5.50 per hundred, that is, when the 30 percent spelter produced is placed on the market and the remaining 70 percent of the zinc in the form of an 80 percent product is shipped to zinc smelters on an ore basis treatment charge. Greater economies could be effected if the electric smelter had retorts of its own to treat this high-grade concentrate of blue powder and zinc oxide.

If a straight zinc concentrate assaying 50 percent zinc is shipped to a zinc smelter, the probable basis of settlement would be as follows: 85 percent of the zinc content at market price of spelter, with $16.00 deducted for treatment charge. With spelter at 5.5 cents per pound, one ton of 50 percent concentrate is worth $30.75.

The electric smelter, after deducting $12.67 for treatment, would return for the same concentrate $23.41. But if a complex zinc concentrate containing approximately 10 percent iron with copper, gold and silver values were to be considered, the difference would be greatly in favor of electric smelting. The penalties on iron, etc., and the poor extraction of copper, gold and silver by the retort smelters would make it impossible for them to compete with the electric zinc smelter.

In the West complex zinc ores are abundant, and in some cases water power can be obtained near the mines, which would be an added advantage to the electric smelter by effecting a saving to the miner on freight on concentrates.

Another probable source of increased revenue would be the sale of zinc products, as zinc oxide, for the paint industry. Statistics show that, in 1910, 16 percent of all zinc produced in the United States was sold as zinc oxide, and in this form brought 0.1 of a cent more per pound than if sold as spelter. There is absolutely no difficulty in making zinc oxide suitable for paint by the electric furnace. The principal accessory required is a bag house and packing plant. A small percentage of the product could be sold as blue powder at prices about equal to that of spelter.

The Rocky Mountain States of the West present great opportunities for the treatment of complex zinc ores by the electric

furnace. As the process stands today it should be a big success on complex zinc ores.

The electric zinc furnace is young, and in the course of operations many improvements will be made and costs greatly reduced.

DISCUSSION.

L. B. Skinner: On page 229 the author says: "The principle used in the elimination of zinc from slags was to make slags of such high formation temperature as to practically volatilize most of the zinc before slagging of the charge begins." I might say that the success of any process for driving zinc out of the slag-forming mixture is almost dependent upon this idea. If the zinc has once been combined in a slag, it is very difficult to rid the slag of it, and that is a very good point to be taken note of in forming the slags for any zinc furnace work.

E. J. Ericson: The author says, on page 229: "If the concentrates are high in iron and low in silica, then a basic slag high in iron is made, and consequently a basic furnace lining (such as magnesite brick) is used; and if the reverse is the case, then high silica slag with silicious furnace linings are used." This changing from basic to acid linings and *vice versa* would cause great difficulties in practice, since, if the two refractories got mixed, contamination would result and the refractory properties be destroyed.

On page 231 the author states his belief that lead can be reduced first from the charge at a lower temperature than that which is required for zinc, and that practically a separation could be effected. This does not seem feasible to me, but it would be interesting to know if he has conducted experiments to justify his conclusion in this particular respect.

W. M. Snow: We all know that the human body is developed without the aid of chemical, mechanical and electrical experts, and we all know it is a very efficient machine; and yet, because the present retort method has developed regardless of scientific experts, many are ready to stamp it as scientifically barbarous.

Industrial evolution without expert advice goes against the delicate sensibilities of some people, but I think if they compare the thermal efficiency of the present condenser with the application of the condenser in the electric furnace, they cannot stray very far, because in the passage of zinc vapor thru the present condenser the dissipation of heat is such that the thermal efficiency is very high, as the recovery of metal by the present retort method is from 86 to 91 percent of the metallic content of the charge, and then not all the zinc in the charge is driven off. In the case of the electric furnace, the method of application of energy has received the greatest attention and the method of condensation the least attention, as the development of electric furnace construction will show.

Mr. Skinner will bear me out that most successful retort furnaces have been built by men who claimed to know nothing about thermochemical conditions, developing furnaces by cut-and-try methods and the aid of knowledge gained by past experiences, and, altho the reduction of zinc ore and formation of zinc vapor in the electric furnace is being followed along scientific lines, one of the most vital points, the condensation of zinc vapor, is trailing along by the cut-and-try method.

J. W. Richards: The ordinary zinc retort runs discontinuously, the composition of the gases changes from the beginning to the end of the heat, and the ordinary condenser has been developed by cut-and-try methods as best adapted to that method of working the ore. The electric furnace working continuously gives a more constant composition of gas, and the condenser which is best adapted to the retort method may not be adapted to the handling of gases in the other method; yet I think the experimenters are going to have a long run if they adopt the cut-and-try method.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Denver, Col., September 9, 1913, President E. F. Roeber in the Chair.

THE ELECTROLYSIS OF CYANIDE SOLUTIONS

By Edward F. Kern.

The experiments upon which this article is based were conducted by Mr. E. H. Koenig and Mr. S. E. Woodworth in the Metallurgical Laboratory of the School of Mines of Columbia University. The work was undertaken with the idea of determining the validity of some of the conflicting statements which were found in reviewing the literature on the electrolysis of cyanide solutions.

The electrolysis of cyanide solutions which are used for the treatment of gold and silver solutions has caused much speculation, not only on account of the application of electrolysis as a means of recovering the gold and silver from leach solutions, but also as a means of regeneration of the cyanide. Two of the main items of expense of the modern cyanide process are the consumption of cyanide and the cost of recovering the gold and silver in a fairly pure state.

Electrometallurgists and electrochemists, having in mind the reduction in cost of treatment of gold and silver ores, have developed a number of processes in which electrolysis has been applied for the recovery of metals from leach solutions and for the regeneration of the cyanide, which during the treatment has been converted into cyanide compounds whose solutions do not dissolve gold and silver.

REVIEW OF LITERATURE.

The following review of the literature gives the basic principles as embodied in the published electrocyanide processes for the treatment of gold and silver ores.

The Siemens and Halske process (Borchers and McMillan, "Electric Smelting and Refining," 1904 Ed., 351; Thompson's "Applied Electrochemistry," 1911 Ed., 63; Kershaw, "Electro-

metallurgy," 1908 Ed., 62) embodies the electro-precipitation of cyanide leach solutions by employing current at low density (0.05 amp. per square foot of cathode surface; 0.54 amp. per square meter) and using iron anodes and lead foil cathodes. The voltage required was between 4 and 7 volts with the electrodes two inches (5.1 cm.) apart. The anodes during electrolysis slowly dissolved, causing the chemical precipitation of iron hydroxide and prussian blue. When the process was first used the anodes were covered with cloth of open texture in order to prevent short-circuiting of the electrodes by the precipitate. Later practice was not to cover the anodes, but to place them further from the cathodes, which permitted the precipitate to fall to the bottom of the vat. The iron anodes have been found to be unsatisfactory on account of the formation of the chemical precipitate, which not only caused short-circuiting of the electrodes, but also caused large consumption of cyanide and carried some gold and silver with it. The consumption of the iron anodes varied from 0.3 to 0.7 pound (0.14 to 0.32 kg.) per ton of solution treated. The gold and silver were finally recovered by melting and cupelling the lead foil cathodes and by smelting of the iron precipitate.

Butters modified the Siemens and Halske process by using peroxidized lead anodes and tin-plate cathodes and employing a higher current density at the cathodes, so that the gold and silver would be deposited as loosely adherent sludge on the cathodes, from which it could be readily removed by scrapers (Thompson's "Applied Electrochemistry," 1911 Ed., 63). The current density was varied from 0.25 to 0.6 ampere per square foot (2.7 to 6.5 amp. per square meter) of cathode surface, and the E. M. F. was about 5 volts. The precipitate was collected at regular intervals and smelted to Doré bullion. The peroxidizing of the lead anodes was accomplished by first scratch-brushing to expose a fresh surface of metal, then making them anodes in an electrolyte containing over 1 percent of potassium permanganate, and electrolyzing with current density of one ampere per square foot for about an hour. The peroxidized lead anodes were found to be insoluble, and to last more than a year with constant use, also less consumption of cyanide resulted than by use of iron anodes.

Butters and other experimenters found that when a low current density at the cathode was used, the metal was deposited

as an adherent, hard film, whereas high current density caused the deposit to form as a non-adherent, pulverulent mass, or as sludge. The amount of metal in solution and the circulation also affect the character of the deposited metal. The smaller the metal content of the electrolyte and the more sluggish its circulation, the less adherent and the more pulverulent the deposit on the cathode. With a very small metal content, and no circulation, the deposit will form as non-adherent sludge, even though a very low current density is employed. The E. M. F. required for the electrolysis depends upon the concentration of the electrolyte and the current density employed.

Other electrolytic cyanide processes, similar in principle to that of Siemens and Halske, have been patented. Most of them differ in that amalgamated plates, or a bath of mercury, are used as the cathodes on which the gold and silver are deposited and form an amalgam. The current density recommended is about 0.5 ampere per square foot (5.4 amp. per square meter) of cathode surface. Use was made of anodes of either peroxidized lead or carbon; the former are the more satisfactory (Borchers and McMillan, "Electric Smelting and Refining," 1904 Ed., 334 to 336).

An advantage claimed for the amalgamated cathodes is that the refining of the deposited metal may be done at lower cost and with less losses than in case solid cathodes are used. Also the amalgamated cathodes permit the electrolysis being conducted in the leaching vats, as in this case the scouring effect of the particles of ore on the deposited metal does not remove the gold and silver from the amalgam. The "clean-up" is accomplished by collecting the amalgam, retorting it, using the distilled mercury for amalgamating fresh plates, and refining the retort bullion to Doré bullion. The addition of sodium chloride to the electrolyte is made in some cases in order to increase the conductivity, thereby causing a reduction in power required for the electrolysis. One of the objections to using amalgamated cathodes is the need of large quantities of mercury.

Parkes devised an electrocyanide process for which a specially designed vat is required (Engineering and Mining Journal, **94**, 263 and 862, 1912). The cathodes are amalgamated copper plates, and the anodes are cast iron. Compressed air at about

12 pounds per square inch (0.8 kg. per sq. cm.) is supplied to the vat for the double purpose of agitating the pulp and to provide oxygen in order to accomplish more rapid dissolving of the gold and silver during the simultaneous leaching and electro-deposition of the metals on the amalgamated cathode. The electrolyte is a dilute solution of potassium or sodium cyanide to which sodium chloride is added. The electrolysis is conducted at 10 to 12 volts between the electrodes, at which potential the inventor claims that sodium hydroxide is formed at the cathode, supplying alkali to the solution, and the chlorine which is liberated at the anodes probably forms chloro-cyanide. Claims are made that the mercury losses are small and that the cyanide consumption per ton of ore treated is about one pound.

Some of the advantages which are claimed for the electrolytic precipitation of cyanide solutions as compared with the precipitations by means of metallic zinc are: (a) The process is suitable for solutions which contain copper; (b) There are less losses during the "clean-up"; (c) The process can be applied to any strength of solution; (d) It is especially applicable to silver cyanide solution; (e) Solutions are not "fouled" during the precipitation; (f) Regeneration of the cyanide is accomplished.

Some of the more recently published electro-cyanide processes apply electrolysis as a means of supplying oxygen to the leach solutions, claiming thereby to enable more rapid and more efficient extraction of gold and silver from ordinary refractory ores. Morris Green studied the action of oxidizers in cyaniding, and his conclusions are that the oxidizers are beneficial in case the ore contains reducing matter (Journ. of Chem., Metal., and Min. Soc. of South Africa, **13**, 355 to 358 (1913). The experiments of Julian are also referred to, the results of which show that free oxygen in cyanide solutions does not play a primary part in the dissolving of gold, but exerts a retarding influence; it does, however, have a secondary effect in the process of dissolving by oxidizing reducing matter, and combining with the occluded hydrogen which is produced through the action of local "voltaic circuits." The equation which represents the dissolving of gold is: $2Au + 4KCN + 2H_2O = 2KAu(CN)_2 + 2KOH + H_2$. The function of oxygen, or oxidizers, in cyaniding ores is to combine with the hydrogen which is liberated during the above reaction.

Mr. Green states that potassium permanganate was used on the Rand as an aid to cyaniding when the ore contained reducing matter. The addition of permanganate, but not in excess, was found to be useful not so much as an oxidizing agent for the reducing matter in the ore, but in that it yielded a solution which dissolved the gold content of the ore more readily. He states that the better manner in which to use permanganate is to employ it as a solution which is brought in contact with the ore to oxidize the reducers, then add the cyanide solution.

With reference to potassium ferricyanide, it is cited that this reagent, which is a well-known oxidizer, has no action upon gold. The addition of potassium ferricyanide to cyanide solutions accelerates the rate of solution of the gold; it, however, destroys potassium cyanide and introduces a reducing agent into the solution, principally ferrocyanide and some cyanate, according to the equation: $2K_3Fe(CN)_6 + 3KCN + H_2O = 2K_4Fe(CN)_6 + KCNO + 2HCN$. Pure potassium cyanate (KCNO) does not effect the dissolution of gold; it is a neutral chemical with reference to potassium cyanide and to gold and silver.

F. H. Aldridge, Jr., utilized electrolysis for supplying the necessary oxygen to cyanide solutions for the treatment of a refractory gold ore which contained amorphous iron sulphide and an excessive amount of reducing matter. (Bull. Am. Inst. Min. Engs., Feb. 12, 1912, p. 177). He stated that the necessary oxygen, according to the following equation, is usually supplied through the medium of air dissolved by the cyanide solution or by the addition of chemical oxidizers: $4Au + 8KCN + 4H_2O + O_2 = 4KAu(CN)_2 + 4KOH + 2H_2O$.

On account of the cost of chemical oxidizers, Mr. Aldridge designed an electrolytic apparatus for supplying oxygen to cyanide solutions, and succeeded in obtaining an extraction by its use which was not possible by the ordinary method of cyaniding. The ore was ground to pass through a 200 mesh screen, mixed with cyanide solution and the pulp agitated by compressed air, and simultaneously electrolyzed, using lead electrodes. This method was found to be a cheaper means of supplying oxygen than by the addition of chemical oxidizers; besides, the electrolysis seemed to regenerate cyanide. The time of electrolysis was from two to four hours. The current density and the potential were not given.

Experiments conducted by Mr. Aldridge showed that alternating current was as suitable as direct current, and it had the additional advantage of giving no deposit of metal on the electrodes. The electrolyzed solution is said to have been especially effective when lead oxide, or lead acetate, was added to it during the grinding of the ore in the tube-mill. Moreover, the electrolyzed solution showed no sulphocyanides when ores which contained sulphides were treated. The electrolysis seemed to have decomposed sulphocyanides and soluble metallic sulphides, but did not affect alkali sulphides.

Over 25,000 tons of oxidized sulphide ores, containing gold, have been successfully treated in the Aldridge apparatus; the cyanide consumption was from one quarter to one half pound (0.11–0.22 kg.) per ton of ore treated, whereas the consumption by the ordinary treatment was about one pound (0.45 kg.) of cyanide per ton of ore. Comparative tests were made, first by electrolyzing the barren sump solution before treatment of the ore, then by conducting the electrolysis with the ore in the solution. The results of these tests showed that the extraction and the cyanide consumption were in each case practically the same.

H. C. Hargrave, in an article on "Cyaniding at the Nova Scotia Mill," gives results of experiments on methods of treating a very refractory silver ore, which contained 6.5 percent iron, 1.1 percent sulphur, 3.4 percent arsenic, 1 percent antimony, 0.9 percent nickel and cobalt, 0.14 percent copper, and 287 ounces of silver per ton. (Eng. and Min. Jour., **93**, 1133 (1912). Hydrogen peroxide, and chemically generated oxygen, were first used with cyanide solutions to overcome the effect of reducing matter in the ore, each giving satisfactory extraction of the silver. Experiments were later conducted in supplying oxygen to cyanide solutions by electrolysis, which showed better results than were obtained by the use of either hydrogen peroxide or chemically generated oxygen. The electrolysis was conducted with direct current, using lead electrodes in some cases, and graphite anodes and iron cathodes in other cases. The graphite anodes gave as satisfactory results as lead anodes. In all cases, the electrolytic method of supplying oxygen gave better extraction of the silver and with less consumption of cyanide than when chemical oxidizers were used. Alternating current at 60 cycles per second was

tried for the electrolysis, but no oxidizing action resulted, probably on account of the frequency being too high.

John C. Clancy presented a paper before the American Electrochemical Society in New York in 1911, entitled "The Clancy Electrochemical Cyanide Process" in which the statement was made that straight cyaniding of a raw refractory ore, similar to that of the Cripple Creek district, yielded but a trifle over 60 percent extraction. (Trans. Am. Electrochem. Soc. **19**, 137–158 (1911). Ores of this character generally have their values associated with compounds in chemical combination, which are not susceptible to the action of solvents until roasting or some other method of oxidation has dissociated the said compounds and liberated the precious metals. Roasting is by all means the cheapest oxidation process so far known; any attempt to oxidize the base constituents by other chemical means is obviously of a very expensive nature, therefore commercially impracticable.

The object of Mr. Clancy's first experiments on Cripple Creek ores was to find a cheap alkaline solvent for telluride of gold, which this ore contains. He discovered that alkali hypochlorites ($NaClO$), hypobromites ($NaBrO$), and hypoiodites ($NaIO$), readily dissolve tellurium, according to the reaction: $Te_2 + 4NaClO + 2H_2O = 2H_2TeO_3 + 4NaCl$.

A preliminary treatment of Cripple Creek ore with a solution of sodium hypochlorite, followed by cyaniding, gave an extraction of 80 to 85 percent of the gold content. The excess of the hypochlorite had to be removed by washing, before adding the cyanide solution, as hypochlorite destroys cyanide. This method of treatment is not sufficiently economical for a commercial process.

Ammonium persulphate ($(NH_4)_2S_2O_8$) acted similarly to sodium hypochlorite in dissolving tellurium, and possesses an advantage in that sulphides of iron are not appreciably attacked by it, and also it does not destroy cyanide, but hastens the dissolving of the gold: $4KCN + 2Au + (NH_4)_2S_2O_8 = 2KAu(CN)_2 + (NH_4)_2SO_4 + K_2SO_4$. The high cost of ammonium persulphate prohibits its use for this purpose.

On account of the high cost of ammonium persulphate, the use of ozone as an oxidizing agent was suggested and tried. It was generated by an electric spark apparatus. It was found that

the cyanide consumption was very high, unless sulphocyanide was used as a reducing agent to destroy the excess of ozone, as expressed by the equation: $3O_3 + KCNS + H_2O = KCN + H_2SO_4$.

The study of a great number of experiments of this character, and of the reactions, suggested that electric current could be utilized as a means of producing the necessary oxidation, while the solution is in contact with the ore, and for generating active solvents of gold and silver from inert compounds.

The Clancy electro-cyanide process, which was the outcome of these experiments, is the employment of electric current to dissociate inert chemicals into active ones, and to regenerate used leach solutions for leaching a fresh lot of ore. A number of different kinds of anodes were tried (lead peroxide, carbon, graphite, iron, and fused iron oxide). The lead peroxide anodes disintegrated; graphite and carbon anodes were satisfactory at low current density, but disintegrated at high current density; the iron anodes dissolved; whereas anodes made by fusing iron oxide in an electric furnace and casting into plates were very satisfactory. The electrolytes which were used contained potassium cyanide, sulphocyanide and a soluble iodide or bromide. The electrolytes were kept neutral during the electrolysis so as to allow the formation of iodo-cyanide (ICN) or bromo-cyanide (BrCN) in contact with the refractory ore. It was found that cyanate, which is an inert chemical, was formed during the electrolysis of a pure cyanide solution.

The electrolysis of a solution of potassium cyanate (KCNO) containing urea produced a solution which dissolved gold. Calcium cyanamide ($CaCN_2$) with cyanate acted similarly to the solution which contained urea and cyanate, giving a solution which on electrolysis at elevated temperatures dissolved gold. It was also found that when a solution of calcium cyanamide was mixed with a solution of alkali ferrocyanide ($K_4Fe(CN)_6$) and allowed to stand for a few hours, it became an active solvent for gold, and remained so even after weeks of standing. Electrolysis of a solution of calcium cyanamide and an alkali ferrocyanide produced an energetic solvent for gold. The electrolysis of a solution containing calcium cyanamide and an alkali sulphocyanide produced cyanide.

The Clancy process as finally adopted, and for which U. S. Patent No. 998,749 was granted, is as follows: The ore is crushed to pass through 100-mesh screen, stirred into a cyanide solution which contains calcium cyanamide, sulphocyanide and an iodide, and the pulp then electrolyzed and simultaneously agitated until the gold has been taken into solution. The solutions which were found to work satisfactory are the following: Solution A: 1 pound (0.45 kg.) cyanide, 2 pounds (0.9 kg.) sulphocyanide, 2 pounds (0.9 kg.) calcium cyanamide, ½ pound (0.22 kg.) iodide and 20 pounds (9.1 kg.) of sodium chloride per ton of water. Solution B: 2 pounds (0.9 kg.) sulphocyanide, 1 pound (0.45 kg.) cyanide, 1 pound (0.45 kg.) iodide and 2 pounds (0.9 kg.) caustic soda per ton of ore. Solution C: 2 pounds (0.9 kg.) sulphocyanide, 1 pound (0.45 kg.) iodide and 2 pounds (0.9 kg.) caustic soda per ton of ore. Used 2 tons of solution per ton of ore. A "protective" alkalinity of from 0.1 to 0.2 pound (0.05 to 0.10 kg.) per ton of solution was maintained by the proper addition of lime. The addition of 20 pounds (9.1 kg.) of sodium chloride to solution A was for the purpose of increasing the conductivity of the solution so that the electrolysis could be conducted with 5 to 6 volts. The current required is from 50 to 120 amperes per ton of ore under treatment per day, supplied at current density of about 50 amperes per square foot (540 amp. per square meter) of anode surface, when using the fused iron oxide anodes. An electrode 3 feet (1 meter) long and 3 inches (7.7 cm.) diameter suffices for the treatment of 3 to 4 tons of ore per day. The electrolyzing tank was made of sheet iron, which also served as the cathode. About eight hours of treatment under electrolysis was found to be sufficient for efficient extraction.

The regeneration of cyanide solution by the Clancy process was accomplished by electrolyzing it for about two hours, first having added 1 pound (0.45 kg.) of protective alkali per ton of solution, which brought the cyanide content up to from 0.5 to 0.6 pound (0.22 to 0.27 kg.) of cyanide per ton of solution. The regeneration would not take place in the presence of a halogen compound unless the solution, which contained sulphocyanide and cyanamide, was alkaline. The whole value of the process depends upon the recovery of the halogen compound, as the

sulphocyanide and the cyanamide are alone decomposed. The loss of cyanide worked out about 1 pound (0.45 kg.) per ton of ore treated, which was regenerated by electrolysis after the addition of cyanamide.

The precipitation of gold on the cathode during the electrolytic treatment of the ore was negligible, as the gold was dissolved as soon as the current was stopped, it being deposited in a very loose, powdery form, which was brushed from the cathode by the agitation of the pulp during the treatment (Trans. Am. Electrochem. Soc., **19**, 382 (1911)).

The principal objections which were advanced against the electric precipitation and recovery of gold and silver from cyanide leach solutions are: (*a*) The need of a large cathode surface and large electrolyzing tanks; (*b*) decomposition of the cyanide solution by electrolysis; (*c*) electrode difficulties; (*d*) scouring of the precipitated metal from the cathodes in case the leaching and the electrolysis are simultaneously carried on in the same tank; (*e*) not so cheap a method of precipitating gold and silver from cyanide leach solutions as by means of either zinc or aluminium.

Epitome of Literature.

The conclusions, which may be drawn by making a digest of the literature on the electrolysis of cyanide solutions, are as follows:

1. Electrolysis has, under favorable conditions, been utilized as a means of precipitating and recovering gold and silver from cyanide leach solutions by electrolyzing the solution with suitable electrodes and with current density of 0.01 to 0.6 ampere per square foot (0.11 to 6.5 amp. per square meter). The metal was deposited as a dense film when the current density was properly regulated with regard to the composition, the concentration and the circulation of the solution. The deposit formed is pulverulent and non-adherent if the solution is dilute, the circulation sluggish, and the current density high.

2. Iron anodes dissolve in cyanide electrolytes causing consumption of cyanide and contamination of the solution by precipitates of iron compounds.

3. Carbon and graphite anodes are satisfactory in cyanide

electrolytes when used at low current density, but they disintegrate at high current density, causing "fouling" of the solution.

4. Peroxidized lead anodes are not dissolved during electrolysis of cyanide solutions, and the consumption of cyanide by their use is less than when metallic iron anodes are employed.

5. Fused iron oxide anodes are permanent in cyanide electrolytes, and the cyanide consumption by their use is small.

6. Oxygen, supplied to cyanide leach solutions either by additions of chemicals or by electrolysis, is beneficial for the treatment of certain refractory ores, in that the extraction is thereby accomplished more rapidly and more efficiently. Electrolysis is claimed to be a cheaper and more efficient method of supplying oxygen to cyanide leach solutions than by the addition of chemical oxidizers.

7. Regeneration of cyanide "work" solutions, which contain cyanogen compounds, is possible by electrolysis. There is controversy as to whether alternating current can be used for this purpose.

8. Electrolysis of solutions containing sulphocyanide and cyanamide by means of direct current at high current density (50 amperes per square foot, 540 amperes per square meter) produced solutions of cyanide. Fused iron oxide anodes were found to be the most satisfactory for this purpose.

9. Electrolysis of solutions containing only cyanide produces cyanates, which will not dissolve gold and silver.

10. Telluride gold ores were effectively treated by use of solutions which contained an halogen-cyanide compound, which was generated by electrolysis of solutions containing an halide salt and cyanogen compounds, employing direct current at high current density for producing the active halogen-cyanide compound.

EXPERIMENTAL PART.

The object in view, when the experiments which are outlined on the following pages were undertaken, was to determine:

1. Which kind of anodes are the most permanent, and which kind give the highest efficiency as regards the regeneration of cyanide solutions.

2. Whether the electrolysis of cyanide leach solutions is

beneficial in increasing the extraction of gold and silver from ores, and in reducing the cyanide consumption.

3. Whether high or low current density produces the greatest amount of "active" solvents in cyanide leach solutions; the "active" solvents being those which are active in dissolving gold and silver.

4. Whether a relative difference in current density at the anode and the cathode affects the consumption of cyanide, and what effect this relative difference in current density has upon the production of "active" solvents in cyanide solutions.

5. What effect the alkalinity of cyanide leach solutions has upon the results of electrolysis.

6. Whether electrolysis of cyanide leach solutions is beneficial in reducing the time required for the efficient treatment of gold and silver ores.

7. What current density at the anode and at the cathode is most suitable for the regeneration of cyanide leach solutions which contain ferrocyanide and sulphocyanide.

Part I. Permanence of Anodes in Cyanide Electrolytes.

This series of experiments was conducted for the purpose of determining whether lead, peroxidized lead, nickel, iron, or oxidized iron is the more suitable for anodes in cyanide electrolytes. The experiments were conducted by electrolyzing five liters of dilute cyanide solution. The cells which contained the electrolyte were square glass aquariums, 11 inches deep, 6 inches wide and 10 inches long (28 x 15 x 25 cm.). The electrodes were 5½ inches (14 cm.) wide and dipped 5 inches (12.7 cm.) into the electrolyte, surface area of both sides 55 square inches (355 sq. cm.). Two anodes and three cathodes were used, the anodes being intermediate to the cathodes, so that both faces of the anodes were exposed to electrolytic action; one cathode was between the anodes and the other two were opposite the outer faces of the anodes. Space between electrodes about 2½ inches (6.3 cm.). The terminals of the two anodes were connected together, and also those of the three cathodes, so that the surface of the anode, and of the cathode, exposed to electrolytic action, were respectively 110 square inches (710 sq. cm.).

The first tests were made by connecting three cells in series:

Cell No. 1 contained two sheet lead anodes and three sheet lead cathodes; cell No. 2 contained two sheet iron anodes and three sheet lead cathodes; cell No. 3 contained two sheet nickel anodes and three sheet lead cathodes. The electrolysis was conducted for two hours, starting with the solution at room temperature. The cyanide content of the solutions was determined before and after the electrolysis by titrating a sample with a standard silver nitrate solution, containing 6.523 grams of silver nitrate per liter, 1.0 c.c. of which was equivalent to 0.005 gram of potassium cyanide. A solution of potassium iodide (1 gram of potassium iodide and 3 grams of potassium hydroxide per 100 c.c.) was used as an indicator for the end-point.

Table I shows the data and the result of the electrolysis on the cyanide consumption.

TABLE I.

Time of Electrolysis, 2 Hours.

Cell	Anodes	C. D. / Sq. Ft. Anode and Cathode	Volts	Percent. KCN		KCN Loss
				Before	After	
No. 1	Lead	10 Amperes	9.5	0.128	0.120	6.25%
No. 2	Iron	10 "	13.0	0.128	0.005	96.0
No. 3	Nickel	10 "	13.0	0.130	0.003	97.5

Immediately upon sending the current through the cells there was a violent evolution of gas at the cathodes, and almost instantly a scum formed around the anodes and colored the solutions; that in cell No. 1 became white, in cell No. 2 became brown, and in cell No. 3 became green. After a short time a thick layer of precipitate deposited on the bottom of each cell. These precipitates were non-conductors, and affected the voltage required for the electrolysis, as the anodes became coated with the precipitate. Upon stirring the electrolytes the precipitate was removed, and the voltage consequently dropped. The voltage readings recorded in the table are those taken directly after stirring the solutions.

The precipitates were collected, washed, and qualitative tests made in order to determine their composition. These tests showed that the precipitate which formed in cell No. 1 was lead

hydroxide, as no cyanogen radical was present, and that those which formed in cells No. 2 and No. 3 were, respectively, a mixture of hydroxides and cyanogen compounds of iron and nickel. The high cyanide consumption in cells No. 2 and No. 3 is accounted for by the formation of the cyanogen precipitates in these cells. The comparative volume of the precipitates in cells No. 2 and No. 3 was about the same, whereas that in cell No. 1 was very much less. On examining the anodes it was found that those in cell No. 1 were not much corroded, and those in cells No. 2 and No. 3 were very much corroded.

The next tests were run similarly to those described above, but in this case the current density was 5 amperes per square foot (54 amp. per square meter) of anode and of cathode surface. The results of these tests are given in Table II.

TABLE II.

Time of Electrolysis, 2¾ Hours.

Cell	Anodes	C. D. / Sq. Ft. Anode and Cathode	Volts	Percent. KCN		KCN Loss
				Before	After	
No. 1	Lead	5 Amperes	9.0	0.210	0.153	27%
No. 2	Iron	5 "	12.5	0.212	0.117	45 "
No. 3	Nickel	5 "	10.5	0.204	0.020	90

Evolution of hydrogen at the cathodes in each of the cells occurred during the passage of the current, and also precipitates formed at the anodes in each case, similar to that which was produced when the electrolysis was conducted at 10 amperes per square foot of electrode surfaces. The precipitate in cell No. 1 was white, that in cell No. 2 was brown, and that in cell No. 3 was green. The lead precipitate was lead hydroxide, the iron precipitate was a mixture of hydroxide and a cyanogen compound, and the nickel precipitate was also a mixture of hydroxide and a cyanogen compound. The voltage readings, which are recorded in the table, were taken directly after stirring the solution to remove the precipitate from the anode.

The results of this series of experiments indicate that lead anodes are corroded relatively more when the current density is low than when it is high. This, no doubt, was due to the lead

being peroxidized at the higher current density, the peroxidation preventing its corrosion during the electrolysis. The iron anodes were corroded relatively less at the lower current density than at the higher. In the case of nickel anodes a variation in the current density did not seem to have any effect on retarding their corrosion in cyanide electrolytes.

It was evident from the results of these tests that anodes of metallic lead, iron or nickel are not suitable for the electrolysis of cyanide solutions, so it was suggested that peroxidized lead and "passive" iron be tried for this purpose. The lead anodes were peroxidized by first scraping them clean, then making them anodes in a dilute (20 percent by weight) sulphuric acid electrolyte, and passing direct current at current density of 10 amperes per square foot (107 amp. per square meter) for about two hours. The iron anodes were rendered "passive" by first removing grease by digesting in hot caustic soda solution, rinsing in water, then putting them into a dilute solution of mixed sulphuric and hydrochloric acids in order to remove all oxide, washing with clean water, thoroughly drying, and finally placing them in concentrated nitric acid and allowing to remain for about two hours. By this treatment the iron plates were coated with a film of magnetic oxide (Fe_3O_4). On removing the plates from the concentrated nitric acid they were immediately washed with clean water and thoroughly dried to prevent the formation of ferric oxide (Fe_2O_3).

The next tests were merely preliminary runs, which were made in order to determine whether the peroxidized lead and the "passive" iron anodes would or would not be corroded by electrolysis in dilute cyanide solutions. The runs were made in the same manner as those which are recorded in Tables I and II. The current in this case was supplied at 10 amperes per square foot (107 amp. per square meter) of electrode surfaces for about one and a half hours. Both anodes, the peroxidized lead and the "passive" iron, were unattacked by the electrolyte, and also scarcely any precipitate or scum formed around the anodes and in the solution. It was found that the addition of 0.2 gram of potassium hydroxide per 100 c.c. of electrolyte reduced the voltage required from about 18 volts to 8.5 volts in each case, when the electrodes were spaced 2½ inches (6.3 cm.) apart. These

experiments showed conclusively that the prepared anodes were insoluble in cyanide electrolytes and that no cyanide was consumed in the formation of chemical precipitates.

Part II. Electrolysis of Cyanide Solutions with Insoluble Anodes.

Having demonstrated that peroxidized lead anodes and "passive" iron anodes are permanent in dilute cyanide electrolytes, the following series of experiments were then made in order to determine the effect of electrolysis upon the dissolving power of cyanide leach solutions and also to ascertain the effect of a difference in current density at the cathode and at the anode. It was evident that, with no corrosion of the anodes, the cyanide consumption during electrolysis would necessarily be due to the formation of cyanate (KCNO) or other oxy-cyanogen compounds.

In order to determine whether strongly oxidizing or strongly reducing conditions during electrolysis increases the dissolving power of cyanide leach solutions, and also whether these solutions cause the consumption of cyanide, the experiments were made as follows: Series A.—The current density at anode and cathode were the same; cells No. 1 and No. 2 were run at 3 amperes per square foot (32 amp. per square meter) of electrode surfaces, and cells No. 3 and No. 4 at 9 amperes per square foot (97 amp. per square meter) of electrode surfaces. Series B was run with different current densities at the anode and at the cathode; the anodes in cells No. 1 and No. 2 were one-third the area of the cathodes, thus obtaining three times the current density at the anode (9 amperes per square foot) as at the cathode (3 amperes per square foot); the anodes in cells No. 3 and No. 4 had three times the area as that of the cathodes, which produced one-third the current density at the anode (3 amperes per square foot) as at the cathode (9 amperes per square foot). By electrolyzing with a higher current density at the anode than at the cathode, the conditions were made reducing, because at the higher anode current density oxygen is readily liberated, whereas with low cathode current density at the cathode the hydrogen will either be taken into solution or else will react with constituents of the electrolyte. The reverse is true when the current density is relatively greater at the cathode than at the anode, as in this case hydrogen will be liberated at the high cathode current

density, and oxygen which is formed at the low anode current density will either be dissolved by the electrolyte or else will oxidize constituents of the solution. The results of these experiments are recorded in Table III and Table IV. Sheet lead cathodes were used in all of the cells.

Pebble-mill cyanide tests were made on the eight electrolyzed solutions of Series A and B, which were run in parallel with similar tests on unelectrolyzed cyanide leach solutions, with the idea of determining whether electrolysis of cyanide solutions will increase their dissolving power for gold and silver in the treatment of a refractory ore. The ore used for these tests was refractory, as is shown by the very low percentage extraction (13 to 15 percent) by direct cyaniding. It assayed 8.5 ounces of gold and silver per ton, 5.3 percent iron as pyrite, 3.7 percent of lead as galena, and some blende. The percentage extraction was obtained on the assays of the tails, and not on the assays of the solutions. Alkalinity tests on the ore showed that it required 0.007 lb. lime per lb. of ore (7 g. per kg.), or 14 pounds of lime per ton of ore. The charge for each pebble-mill test was: 2½ pounds (1.35 kg.) of ore, which was crushed to pass through an 80-mesh screen, 4½ pounds (5.1 kg.) of silica pebbles (1 to 1½ inches (2.5 to 4 cm.) diameter), 9 grams of lime, and 3 liters of solution containing 0.2 gram of potassium cyanide and 0.5 gram of free potassium hydroxide per 100 c.c. The time of treatment and the results are given in Tables III and IV.

After the treatment of the ore in the pebble mills the pulp was thoroughly stirred and a dip sample immediately poured on the filter in a Buechner suction funnel. The solution which passed through the filter was retained as a sample in which the cyanide content was determined for the purpose of obtaining the cyanide loss; and the pulp which remained on the filter was thoroughly washed, dried, bucked to pass through a 60-mesh screen, and assayed, which gave the percentage extraction of gold and silver.

As far as the electrolysis of the cyanide leach solutions is concerned, the results as shown in Tables III and IV substantiate the hypothesis that a greater loss of cyanide occurs by electrolyzing the solution at higher than at lower current densities, which loss is undoubtedly due to the decomposition of cyanide,

Table III.

Series A—Insoluble Anodes and Sheet Lead Cathodes.

Time of Electrolysis, 2 hours. Time of Pebble-mill Treatment, 21½ hours. Assay of Ore before Treatment, 8.5 ounces Au and Ag per ton.

Cell	Anodes	C. D. per Square Foot		Average voltage	Temp. °C.		Percent KCN		Assay of Tails Au and Ag per ton	Per cent. Extraction	Perct. KCN after leaching	Percentage KCN Loss		
		At Anode	At Cathode		Start	Finish	Start	Finish				After elect'y	Elect'y and leach.	By leach.
No. 1	PbO_2	3 amperes	3 amperes	4.15	19.0	22.5	0.214	0.124	7.26 ozs.	14.5	0.065	42.8	70.0	27.2
No. 2	Fe_3O_4	3 "	3 "	4.0	19.0	21.0	0.214	0.201	6.98 "	17.8	0.130	6.0	40.0	34.0
No. 3	PbO_2	9 "	9 "	7.3	22.0	31.5	0.203	0.100	7.24 "	14.8	0.045	50.0	78.0	28.0
No. 4	Fe_3O_4	9 "	9 "	6.8	21.5	28.5	0.203	0.141	6.96 "	18.0	0.078	30.5	62.0	31.0
Unelectrolyzed leach solution				. . .	. . .	. . .	0.200	. . .	7.16 "	15.8	0.127	. . .	36.5	36.5

Table IV.

Series B—Insoluble Anodes and Sheet Lead Cathodes.

Time of Electrolysis, 2 hours. Time of Pebble-mill Treatment, 45 hours. Assay of Ore before Treatment, 8.5 ounces Au and Ag per ton.

Cell	Anodes	C. D. per Square Foot		Average voltage	Temp. °C.		Percent KCN		Assay of Tails Au and Ag per ton	Per cent. Extraction	Perct. KCN after leaching	Percentage KCN Loss		
		At Anode	At Cathode		Start	Finish	Start	Finish				After elect'y	Elect'y and leach.	By leach.
No. 1	PbO_2	9 amperes	3 amperes	5.25	19.0	24.0	0.216	0.159	7.43 ozs.	12.6	0.061	26.5	72.0	45.5
No. 2	Fe_3O_4	9 "	3 "	5.0	19.0	22.0	0.216	0.200	7.56 "	11.0	0.100	7.0	53.5	46.5
No. 3	PbO_2	3 "	9 "	4.4	19.0	22.0	0.215	0.118	7.24 "	14.8	0.027	45.0	87.0	42.0
No. 4	Fe_3O_4	3 "	9 "	4.7	19.0	22.0	0.215	0.192	7.74 "	8.9	0.094	10.5	56.3	45.8
Unelectrolyzed leach solution				. . .	. . .	. . .	0.200	. . .	7.32 "	13.8	0.127	. . .	47.0	47.0

principally by oxidation with the formation of cyanates and other oxy-cyanogen compounds. In Series B, cells No. 1 and No. 2, in which the same high current density (9 amperes per square foot or 97 amperes per square meter) was maintained at the anode as that which was maintained at both electrodes in cells No. 3 and No. 4 of Series A, but operating under highly reducing conditions at the cathode (3 amperes per square foot or 32 amperes per square meter), the cyanide consumption was much less, which demonstrates that the loss of cyanide during electrolysis is due principally to oxidation with the formation of oxy-cyanogen compounds. The results which were obtained in the runs of Series B, cells No. 3 and No. 4, having the same current density (3 amperes per square foot) at the anodes as cells No. 1 and No. 2 of Series A, but three times the current density at the cathode (9 amperes per square foot), showed greater cyanide consumption, which was additional proof that electrolytic oxidizing conditions are detrimental to cyanide leach solutions..

In each case the "passive" iron anodes showed to better advantage than did the peroxidized lead anodes, in that the cyanide consumption was less during electrolysis. In all cases the cyanide consumption was less and the extraction of gold and silver from the ore was slightly better when the electrolysis was conducted with high current density at the anode and low current density at the cathode, than when the conditions of electrolysis were the reverse.

As far as the pebble-mill leaching tests and the cyanide consumption are concerned, the electrolyzed solutions showed no appreciable advantage over the unelectrolyzed leach solutions, as in all cases the percentage extraction by leaching did not differ greatly. The extraction was in no case increased more than 2 percent, which difference might easily have been due to sampling and assaying discrepancies. Moreover, the actual cyanide consumption during leaching was a little greater in the unelectrolyzed solutions than in the electrolyzed solutions. The total loss of cyanide in the electrolyzed solutions was far greater than that in the unelectrolyzed solution, especially when peroxidized lead anodes were used. The large consumption of cyanide during the electrolysis, and with only a small increase in the extraction in case of the "passive" iron anodes, and no increase in the case

of the peroxidized lead anodes, show that electrolysis of the cyanide leach solutions was not beneficial in obtaining a better extraction of the special ore which was treated.

Part III. Electrolysis of Cyanide Solutions Containing Sulphocyanide and Ferrocyanide.

The two principal products which are formed during the cyanide treatment of gold and silver ores, which contain iron sulphides, and which are produced at the expense of cyanide, are alkali sulphocyanides and ferrocyanides. As the experiments which are recorded in Tables I, II, III and IV showed pretty conclusively that no benefit was attained by the electrolysis of solutions which contained only potassium cyanide, the remaining experiments were run with the idea of determining whether sulphocyanide and ferrocyanide in cyanide leach solutions could be converted into cyanide by means of electrolysis.

The experiments were conducted in the same manner as those described in Part II, using 5 liters of solution which contained a known amount of free potassium cyanide, 0.3 gram of potassium hydroxide and 0.2 gram equivalent of potassium cyanide in the form of either potassium sulphocyanide (0.3 gram) or potassium ferrocyanide (0.19 gram), each per 100 c.c. The experiments were divided into Series A, Series B and Series C, the results of which are recorded in Tables V, VI and VII.

Series A.—The electrolyte of this series contained 0.1 gram of potassium cyanide, 0.3 gram potassium hydroxide and 0.3 gram potassium sulphocyanide per 100 c.c. Cells No. 1, No. 3 and No. 5 contained peroxidized lead anodes and sheet lead cathodes; cells No. 2, No. 4 and No. 6 contained "passive" iron anodes and sheet lead cathodes. The exposed surfaces of the anodes and cathodes in cells No. 1 to No. 4, inclusive, were 110 square inches (710 sq. cm.); the anodes in cells No. 5 and No. 6 had surface exposed to electrolysis 37 square inches (240 sq. cm.), whereas that of the cathodes was 110 square inches. The electrodes in each cell were spaced 2½ inches (6.3 cm.) apart, and the electrolysis conducted at room temperature. Table V contains the data and the results of this series of experiments.

The anodes in cells No. 3 and No. 4, which were run at high current density (9 amperes per square foot or 97 amperes per

square meter) showed corrosion, and the respective solutions contained white and brown precipitates, which accounts for the higher cyanide consumption in these cells. Scarcely any corrosion of the anodes occurred in cells No. 1, No. 2, No. 5 and No. 6, which were run at low current density (3 amperes per square foot, or 32 amperes per square meter). The cyanide consumption in the cells which were run at low current density at the anode indicates that, when sulphocyanide is present in cyanide electrolytes, oxidizing conditions give less consumption of cyanide. When "passive" iron anodes were used, the cyanide loss was smaller than in the case of peroxidized lead anodes.

TABLE V.

Series A—Electrolyte Containing Cyanide and Sulphocyanide.

Cells	Anodes	C.D. per square foot		Aver. Voltage	Percentage KCN			KCN Loss	
		At Anode	At Cathode		Start	After 1 hour	After 2 hours	After 1 hour	After 2 hours
No. 1	PbO_2	3 Amp.	3 Amp.	3.7	0.100	0.082	0.068	18.0%	32.0%
No. 2	Fe_3O_4	3 "	3 "	3.8	0.100	0.092	0.094	8.0	6.0
No. 3	PbO_2	9 "	9 "	6.5	0.100	0.075	0.071	25.0	29.0
No. 4	Fe_3O_4	9 "	9 "	6.0	0.100	0.071	0.011	29.0	89.0
No. 5	PbO_2	3 "	9 "	4.2	0.101	0.083	0.073	17.8	27.7
No. 6	Fe_3O_4	3 "	9 "	4.0	0.101	0.097	0.094	4.0	6.9

Series B.—The electrolytes of this series contained 0.1 gram potassium cyanide, 0.3 gram potassium hydroxide and 0.19 gram of potassium ferrocyanide (equivalent to 0.2 gram potassium cyanide) per 100 c.c. Cells No. 1 and No. 3 contained peroxidized lead anodes and sheet lead cathodes, and cells No. 2 and No. 4 contained "passive" iron anodes and sheet lead cathodes; the surface of the electrodes was 110 square inches (710 sq. cm.). The current supplied to cells No. 1 and No. 2 was one-third the amount which was supplied to cells No. 3 and No. 4; the conditions of electrolysis were otherwise similar. Space between electrodes was 2½ inches (6.3 cm.). The records are given in Table VI.

The anodes in the cells of this series of experiments showed practically no corrosion, showing that the ferrocyanide radical did not dissolve them. The electrolytes in all the cells took on

a deep yellow color during the electrolysis, but no precipitate formed. The cyanide consumption in the case of the "passive" anodes at low current density showed very low cyanide consumption, and at the higher current density the consumption was less than half of that which occurred with the peroxidized lead anodes. The cyanide consumption in these cells which contained ferrocyanide was no doubt due to the formation of oxy-cyanides by direct oxidation of the cyanide at the anodes.

TABLE VI.

Series B—Electrolyte Containing Cyanide and Ferrocyanide.

Cells	Anodes	C.D. per square foot		Aver. Volt.	Percentage KCN			KCN Loss	
		At Anode	At Cathode		Start	After 1 hour	After 2 hours	After 1 hour	After 2 hours
No. 1	PbO_2	3 Amp.	3 Amp.	3.8	0.101	0.078	0.055	22.7%	45.5%
No. 2	Fe_3O_4	3 "	3 "	3.8	0.101	0.101	0.099	0.0	1.9
No. 3	PbO_2	9 "	9 "	8.0	0.101	0.061	0.033	39.6	67.3
No. 4	Fe_3O_4	9 "	9 "	7.0	0.101	0.085	0.070	15.8	30.6

Series C.—The electrolytes of this series of experiments contained approximately 1 gram of free potassium cyanide, 0.3 gram of potassium hydroxide and an equivalent of 0.2 gram of potassium cyanide in the form of either potassium sulphocyanide (cells No. 1 and No. 2) or potassium ferrocyanide (cells No. 3 and No. 4) per 100 c.c. The anodes in cells No. 1 and No. 3 were peroxidized lead, surface exposed to electrolysis 37 square inches (240 sq. cm.), and the anodes in cells No. 2 and No. 4 were "passive" iron, of the same size. The cathodes in each cell were sheet lead, having surface exposed to electrolysis 110 square inches (710 sq. cm.). The electrodes were spaced 2½ inches (6.3 cm.) apart, and the electrolysis conducted at ordinary temperature. The current density, voltage and the results of the electrolysis are given in Table VII.

The results of this series of experiments confirm those of the former experiments. In the first place, "passive" iron anodes were found to be more suitable for the electrolysis of cyanide solutions than peroxidized lead anodes; second, the consumption of cyanide in the case of the "passive" iron anodes was much less than with peroxidized lead anodes; and also, under oxidizing

electrolytic conditions, the sulphocyanide solutions after one hour's electrolysis with "passive" iron anodes showed regeneration to cyanide, and, in case of the ferrocyanide solutions under similar conditions, consumption of cyanide was scarcely noticeable. The presence of either sulphocyanide or ferrocyanide in cyanide electrolytes retards the consumption of cyanide, which is due, no doubt, to these compounds being more readily oxidized than cyanide, therefore acting as a protecting agent during the electrolysis.

TABLE VII.

Series C—Electrolyte Containing Free Cyanide and Combined Cyanide.

Cells	Anodes	C.D. per sq. ft.		Aver. Volt.	0.2 Percent. Equivalent of KCN as	Percentage KCN				KCN Loss		
		At Anode	At Cath.			Start	After 1 Hour	After 2 Hour	After 2½ Hrs.	After 1 Hour	After 2 Hours	After 2½ Hrs.
1	PbO_2	9 amp.	3 amp.	6.0	KCNS	0.99	0.920	0.320	.....	7.0%	67.6%	.....
2	Fe_3O_4	9 "	3 "	5.5	KCNS	0.99	0.705	0.920	.....	28.7	7.0	.....
3	PbO_2	9 "	3 "	6.0	$K_4Fe(CN)_6$	0.99	0.820	0.670	.....	17.0	32.3	.....
4	Fe_3O_4	9 "	3 "	5.5	$K_4Fe(CN)_6$	1.00	0.995	0.980	0.980	0.5	2.0	2.0%

CONCLUSIONS.

A summary of the results of the experiments on the electrolysis of cyanide solutions, which are described under Parts I, II, and III, is as follows:

1. The electrolysis of cyanide solutions by direct current and by means of insoluble anodes is accompanied by progressive consumption of cyanide, which is the result of oxidation of the cyanide to oxy-cyanide compounds.

2. Anodes of metallic iron, nickel and lead are dissolved during the electrolysis of cyanide solutions; these metals on entering the solution are immediately precipitated, the lead as hydroxide, and the iron and nickel as a mixture of hydroxide and cyanogen compounds. The consumption of cyanide by means of these anodes was relatively greater the lower the current density, as at high current density oxygen was simultaneously liberated, and consequently less metal was dissolved. The consumption of cyanide was less in case of the lead anodes than with either the iron or the nickel.

3. Peroxidized lead and "passive" iron anodes were found to be more permanent than metallic anodes of either iron, nickel or lead. There was no apparent corrosion of either the peroxidized lead or the "passive" iron anodes so long as they were kept continuously in use, but exposure to the atmosphere for several days destroyed their permanency, due to spontaneous oxidation. The "passive" iron anodes were found to be more satisfactory than peroxidized lead anodes for electrolyzing cyanide solutions in that the cyanide consumption was much less and the voltage was slightly less in most cases.

4. Electrolysis of cyanide solutions, which were used for leaching a special refractory gold and silver ore containing sulphide minerals, was found not to be beneficial either in reducing the cyanide consumption or increasing the percentage extraction.

5. Low current density at both the anode and the cathode gave less consumption of cyanide, and in several cases seemed to have produced a slight amount of "active" solvents, but not enough to compensate for the loss of cyanide which resulted by the electrolysis.

6. The cyanide consumption during electrolysis of cyanide solutions was greater when the current density at the cathode was run higher than at the anode, which suggested that the loss of cyanide is the result of oxidation, with the formation of oxycyanogen compounds. The lower the current density at the anode and the higher it is at the cathode, relatively greater is the cyanide consumption.

7. When cyanide solutions containing sulphocyanides or ferrocyanides were electrolyzed, the cyanide consumption was much less than that which occurred in pure cyanide solutions, which indicates that sulphocyanide and ferrocyanide act as protective agents during the electrolysis of cyanide solutions.

8. The increased alkalinity of cyanide solutions reduced the cyanide consumption during electrolysis, due to increasing the conductivity of the solution. The higher the voltage the greater the cyanide consumption, and *vice versa.*

9. The electrolysis of cyanide leach solutions had no apparent effect upon reducing the time required for treating a refractory ore nor in increasing the percentage extraction.

10. Peroxidized lead anodes and "passive" iron anodes are corroded in cyanide electrolytes which contain sulphocyanide, if the electrolysis is conducted at high current density. No apparent corrosion occurred when a low current density (below 3 amperes per square foot, or 32 amperes per square meter) was used.

11. "Passive" iron anodes are more satisfactory than peroxidized lead anodes for the electrolysis of cyanide solutions which contain either sulphocyanides or ferrocyanides in that by their use the cyanide consumption is very much less and the voltage slightly less. The lower the current density the smaller the consumption.

12. The regeneration of cyanide solutions which contain sulphocyanide and ferrocyanide does not occur by electrolysis with direct current, whether the conditions of electrolysis be made oxidizing or reducing by varying the relative current densities at the anode and the cathode.

The suggestions which were stimulated by the foregoing experiments will form the basis of a future series of experiments, which it is hoped will contribute something more to the knowledge of what occurs during the electrolysis of cyanide solutions. It is also hoped that the discussion will bring out other suggestions of conducting the work and also contribute the results of unpublished research in this interesting field.

Metallurgical Laboratory,
School of Mines of Columbia University,
New York City, July, 1913.

DISCUSSION.

G. H. Clevenger: It is an extremely dangerous thing to form conclusions regarding the life of anodes from small scale experiments, and particularly small scale experiments which cover a limited period of time. During an engagement covering some three or four years with Mr. Charles Butters, as metallurgical engineer, I had a splendid opportunity of observing and studying the electrolytic precipitation of gold, silver and copper from

cyanide solutions as practiced at Virginia City, Nevada; Minas Prietas, Mexico, and San Sebastian, Salvador. A great many experiments were made, covering a period of a number of years, for the purpose of finding the most suitable anodes for use under these particular conditions. In the course of this work I think about everything under the sun which gave any promise whatever in this direction was tried, excepting possibly passive iron. It would therefore be of a great deal of interest and possibly of practical value to try passive iron as an anode upon a working scale, not for a period of one day, one week or one month, but for at least a year. It has been our experience that, in order to form any sort of correct conclusions in regard to the life of anodes, it is necessary to test them out over a long period of time. Anodes which appear entirely unaffected for the first few weeks often fail suddenly later. The current densities used at the three plants referred to varied from ¼ ampere to 1 ampere per square foot (3 to 11 amp. per sq. meter) of anode surface. In most cases the cathode surface was approximately the same as the anode surface. At this current density and under the conditions mentioned, peroxidized lead anodes had the longest life of anything tried. In addition to having a longer life than any other electrode which we tried, we found a considerable advantage in the fact that, even after the electrode had served its useful life, there was considerable salvage which could be recovered in the way of lead as well as other by-products, either by shipping or locally. It will be noted that, even with the comparatively low current density used in electrolytic precipitation, peroxidized lead anodes do not have an indefinite life. I suspect that the same would be true of passive iron, for sooner or later the coating which gives it its peculiar properties would fail just as the lead peroxide coating has been found to fail with lead anodes in the course of time in actual practice.

Now, it might appear that a year was a rather long life for an anode, but when actual facts are considered it will be seen that anodes must at least have this life in order to prove at all economical. Taking for example the installation at Virginia City, the total weight of the lead anodes in use was approximately 32 tons; in other words, it required 32 tons of sheet lead to equip the boxes. Now, if these anodes have a life of but a year,

it will be apparent that it was necessary to install every year 32 tons of lead anodes. It will be readily appreciated that this is no small matter, even when allowance is made for the lead recovered from the old anodes.

Our experience with Acheson graphite under these conditions will be interesting. After a time the graphite becomes soft and mushy, and rapidly disintegrates. I am unable to tell why this should happen, but can only say that it does happen in the course of time.

Dr. Kern gives comparisons between electrolyzed and what he terms "leach" solutions. This term "leach" used in this connection is a new one to me, although my experience in the cyanide process is quite extensive in this and other countries. I presume that he means by leach solution what we commonly call pregnant solution, that is, solution which is passed through a charge of sand or through regular slime treatment and has taken up a certain amount of gold and silver. His comparisons would have been far more to the point if he had made them between his electrolyzed solution and solution carrying zinc. I might mention that in present-day cyanide practice, when electrolytic precipitation is not practiced, zinc precipitation is used, and therefore the solutions invariably carry zinc. At Virginia City we sometimes noted better extractions under certain conditions with our solutions, the major part of which had been precipitated electrolytically, than were obtained at certain other mills where zinc precipitation was exclusively used and where the proportion of zinc in solution was greater. It is therefore apparent that it is possible that comparisons between electrolyzed solutions and solutions precipitated by zinc, which are the actual conditions of practice, might have shown up somewhat more favorably for the electrolyzed solutions than the results which he presents. I agree with conclusion 1 of his paper, and in a general way with most of the other conclusions with the exception of 12, although I am of the opinion that he has based certain of his conclusions upon rather insufficient data. In conclusion 12 he states: "The regeneration of cyanide solutions which contain sulphocyanide and ferrocyanide does not occur by electrolysis with direct current, whether the conditions of electrolysis be made oxidizing or reducing by varying the relative current densities at the anode and the cathode."

I have never had occasion except in a superficial way to investigate the electrolysis of ferrocyanide compounds, but the electrolysis of sulphocyanide was the subject of a preliminary investigation made at Minas Prietas, Mexico, a number of years ago. During this work, E. M. Hamilton and myself had found that a preliminary treatment with sodium hydrosulphide was beneficial to the treatment of certain of the complex silver ores of this district by the cyanide process. However, when such treatment was practiced there was a large consumption of cyanide, which was finally traced to the formation of sulphocyanide. Our efforts were then directed toward finding a means of regenerating cyanide from the sulphocyanide, and during this work we found that by electrolysis we could do it to a certain extent. It will be noted that this result is directly contrary to Dr. Kern's statement that cyanide cannot be regenerated from sulphocyanide by electrolysis. Since that time I have made other experiments which confirm these earlier results. Here again the trouble seems to be insufficient data. If Dr. Kern had made up a solution of potassium sulphocyanide containing no cyanate, and had electrolyzed this solution under proper conditions, he would have found that there was a gradual gain in the cyanide strength as indicated by the ordinary Liebig titration. This reaches a certain maximum, and then upon continuing to pass the current through the solution there is a gradual falling off of the cyanide strength as indicated by the titration. This is due to the decomposition of the cyanide formed by the current, as shown in my paper upon the "Electrolysis of Cyanide Solutions." It would appear that in the solution which he electrolyzed, which was a mixture of simple cyanide and sulphocyanide, the decomposition of the cyanide went on at about the same rate as the regeneration of the cyanide from the sulphocyanide. The statement as it stands, to the effect that there is no regeneration of cyanide from sulphocyanide, is certainly inaccurate.

J. W. Richards: Some years ago I was interested in the idea of electrolyzing cyanide solutions, and I precipitated gold from such solutions in a way that I have not seen published, so I will briefly mention it.

I made a small Castner mercury cell with a layer of mercury in the bottom, and placed in one side sodium chloride solution

and suitable carbon anodes for electrolyzing it. I placed in the other side a gold cyanide solution and cathode plates for receiving the gold. On passing the current through and rocking the cell, we had the formation of sodium amalgam on one side and the decomposition of the sodium amalgam on the other side, all the cyanogen brought to the other side re-forming sodium cyanide while the gold was precipitated. These were only laboratory experiments, but the operation is practicable. You can precipitate the gold electrically, using a sodium amalgam anode, so that all the cyanogen brought to the anode forms sodium cyanide and all the gold is precipitated.

I corresponded with Mr. Castner, whose patents covered the use of the apparatus, and he said he was not interested in the matter; and, as this use of the apparatus would have been subsidiary to the Castner patents, I did not go any further with the matter.

G. H. Clevenger: I am very much interested to learn that Dr. Richards tried an experiment of this kind for the precipitation of gold and silver from cyanide solutions. I might mention that one of the great difficulties in connection with the application of any electrolytic process for this purpose, in which mercury is used as the cathode, is the obtaining of sufficient surface with a reasonable weight of mercury. I suspect that this would be one disadvantage of the apparatus as described by Dr. Richards. In other words, it would have a limited capacity.

A number of years ago I witnessed an interesting experiment carried on at Minas Prietas, Mexico, by a young man whose name I do not now recall, who had apparently overcome this difficulty by using a large mass of fine iron wire. He used a cell very similar to that described by Dr. Richards, by means of which he deposited sodium by the electrolysis of salt solution in a bath of mercury in which rotated a perforated cylinder containing fine iron wire. In this way the iron wire was kept covered by a thin layer of sodium amalgam. The solution to be precipitated was caused to pass through the compartment in which this drum rotated. This apparatus appeared at the time to be very promising. For some reason its development was abandoned. Further work along these lines might be productive of interesting and valuable results.

F. A. LIDBURY: Of course, such a method would result in piling up large quantities of caustic with the cyanide.

J. W. RICHARDS: That is not undesirable, as the presence of the caustic would be usually an advantage in the leaching, to neutralize acidity of the ore.

F. C. FRARY: In the eighth conclusion it is evident that the author misstated what he meant to say. From his statement it would appear that the increased alkalinity of cyanide solutions reduced the cyanide consumption and voltage during electrolysis. The hydroxyl ion is liberated at a very low anode potential from such alkaline solutions, so we will not get a high enough potential to discharge the CN ion, and probably not sufficient potential at the anode to cause the oxidation of cyanide to cyanate. The oxidation is dependent on the anode potential at which the oxygen is liberated, and it would appear that what he means by the increase in conductivity is really the decrease in anode potential.

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THE ELECTROLYSIS OF AQUEOUS SOLUTIONS OF THE SIMPLE ALKALINE CYANIDES

By G. Howell Clevenger* and Mortimer L. Hall.†

The constantly recurring proposals for the use of the electric current as an aid to the extraction of gold and silver from refractory ores by the cyanide process, together with the former quite extensive use of electrical precipitation of gold and silver from cyanide solutions, makes of interest a study of the decomposition of the various cyanides by the electric current. A consideration of the whole field is of importance, but the tremendous amount of investigation necessary to thoroughly work out the problem as applying to mill solutions has led us to confine the present paper to, perhaps, the most important single constituent of such solutions—the simple alkaline cyanides. Although other alkaline cyanides are generally present in mill solutions, for the sake of simplicity we have taken the reactions involved with the potassium salt as being more or less representative of the whole class. We have, therefore, used chemically pure potassium cyanide throughout our experimental work.

Electrolysis of Aqueous Solutions of Potassium Cyanide.

Schlagdenhauffen[1] (1863) states that ammonia, carbon dioxide and possibly cyanate are the products of the electrolysis of a cyanide solution. Kalbe[2] states that ammonia, carbon dioxide and cyanate are the products formed by the electrolysis of a cyanide solution. Allen[3] says that the cyanate is formed at the anode by the electrolysis of the corresponding cyanide. Paterno

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[1] Jahresbuch der Chemie, p. 305. (1863)
[2] Liebig's Annalen, **64**, 236.
[3] Commercial Organic Analysis, second edition, London, 3 [3], 481 (1896).

and Pannain,[4] in the course of their work of finding the most favorable conditions for the oxidation of potassium cyanide to cyanate electrolytically, ascertained the following: *First,* a concentration of potassium cyanide of between 4 and 6 gram mols. per liter; *second,* the presence of 1 gram mol. of potassium hydroxide per liter; *third,* a voltage of 4 to 6; *fourth,* a current density at the anode of 9.3 to 37.2 amperes per square foot (100 to 400 amperes per square meter); *fifth,* agitation of the solution at the anode.

A solution containing 72 grams of potassium cyanide, when electrolyzed under the above conditions, yielded 32 grams of the purest cyanate which precipitated out during electrolysis, while 49 grams remained in solution. There also remained in solution 2 grams of undecomposed potassium cyanide. The cyanate produced was 90.3 percent of that theoretically possible from 72 grams of cyanide, and 92.8 percent of the 70 grams of cyanide actually decomposed. When maintaining the above conditions except that the potassium hydroxide was omitted, the solution assumed a reddish-brown color and there was deposited an amorphous brown substance. Even in the presence of potassium hydroxide the solution showed this brown coloration, unless it was agitated at the anode.

Experimental.

The electrolyzing cell used throughout this investigation was made by mounting two plates of Acheson graphite upon wooden supports resting upon the top of a glass battery jar, which held about 1,500 cc. of solution. The distance between the electrodes could be readily varied from ¼ inch to 1 inch (0.6 cm. to 2.5 cm.). The submerged portion of the electrodes measured 1½ inches by 5½ inches (3.8 x 14.0 cm.), giving an area of 8¼ square inches (53.2 sq. cm.) per face.

Experiment I. A solution containing 0.493 percent of potassium cyanide, but containing no protective alkalinity,[5] was

[4] Gazetta Chimica Italiana, 34 [2], 152.

[5] Clennell (The Chemistry of Cyanide Solutions, New York, 1904, p. 61) gives the following definition of protective alkalinity:

"The term 'protective alkali' is based on the assumption that certain ingredients in an ordinary working solution will be wholly or partially neutralized, on addition of a dilute mineral acid, or carbonic acid, before any decomposition of cyanide occurs. In the treatment of ores the addition of lime or caustic soda is made with the object

electrolyzed for a period of three hours and twenty minutes. Samples of the solution were taken at the intervals indicated. The simple cyanide in these was determined by the Liebig titration, using a 10 percent solution of KI as an indicator. The results are given in Table I.

TABLE I.

Total Time Electrolyzed Minutes	Percent KCN	Temperature Degrees Centigrade	Amperes Indicated	Ampere Hours for Interval	Ampere Hours, Cumulative
0	0.493				
20	0.475		1.45	0.48	0.48
33	0.465		1.70	0.34	0.82
47	0.448	25	1.85	0.46	1.28
60	0.433	26	1.95	0.41	1.69
90	0.405	28	2.05	0.98	2.67
110	0.385	29	2.0	0.67	3.33
		Interrupted			
130	0.350	22	1.8	0.60	3.93
160	0.330	25	1.8	0.90	4.83
200	0.285	27.5	1.9	1.27	6.10

The voltage was constant at 5.8.

At the cathode there occurred throughout the experiment a violent ebullition of gas having a slight odor of HCN, but presumably mostly H. A drop of HCl upon a glass rod, held just above where the gas was being evolved, gave a dense white fume, indicating the presence of NH_3. Gas was slowly evolved at the anode, which was undoubtedly largely O with some CO_2.

Inspection of Curve I shows that the cyanide decomposed is directly proportional to the ampere hours of current supplied.

Experiment II. A solution containing 0.49 percent of potassium cyanide and 45 points of protective alkalinity (100 points

of protecting the cyanide from the decomposing effect of various matters contained in the ore, and of the carbonic acid in the air. As a matter of fact a cyanide solution undergoes gradual decomposition, with evolution of hydrocyanic acid, even when an excess of caustic alkali is present, so that the protection afforded is only partial and temporary, and not absolute. There is, however, a fairly definite point in the neutralization of an ordinary solution (containing free cyanide, hydrates, carbonates, etc.), at which the decomposition, with formation of hydrocyanic acid, begins to take place with marked rapidity. In the absence of zinc this point corresponds with tolerable exactness to the alkalinity of the hydrates and carbonates present toward phenol-phthalein, and if the amount of acid corresponding to this alkalinity be added, with agitation, to such a solution, the cyanide strength, as shown by immediate titration with silver nitrate, will remain apparently unchanged. In this case, therefore, protective alkali = hydrate + ½ carbonate."

protective alkalinity corresponds to a saturated solution of lime in water at ordinary temperatures—0.13 percent CaO) was electrolyzed for a period of two hours and thirty minutes with the result given in Table II.

TABLE II.

Total Time Electrolyzed Minutes	Percent KCN	Points Alkalinity	Amperes Indicated	Ampere Hours for Interval	Ampere Hours, Cumulative
0	0.49	45	2.0		
20	0.465	42.5	2.0	0.67	
40	0.44	37.5	2.1	0.70	1.37
60	0.42	27.5	2.0	0.67	2.04
90	0.385	22.5	2.1	1.05	3.09
120	0.345	20.0	2.0	1.00	4.09
150	0.305	15.6	2.0 to 1.0*	0.75	4.84

* Battery ran down.

The products of electrolysis appeared to be the same as in Experiment I except that a white granular precipitate formed which, when separated from the solution by filtration, effervesced with acids and proved to be $CaCO_3$. This confirmed the suspicion that CO_2 was formed during electrolysis. There was a constant fall in protective alkalinity throughout the experiment.

Inspection of Curve II shows that the decomposition of the cyanide is proportional to the ampere hours of current supplied, and practically coincident with Curve I during the first half of its course. It then begins to rise, and terminates somewhat above Curve I.

Having confirmed the statements of other investigators that the alkaline cyanides are decomposed by electrolysis, and having shown in a general way the rate at which decomposition takes place with a given current, both with and without protective alkalinity, the next step became the definite study of the decompositions involved.

Cyanates and Isocyanates. There is considerable confusion in regard to these two series of compounds. This doubtless arises through difference in opinion existing between chemists in regard

to the molecular constitution of the metallic cyanates. Allen[6] says: "The molecular constitution of the metallic cyanates is uncertain. Some authorities, arguing from the fact that potassium cyanate when distilled with potassium ethyl sulphate yields ethyl isocyanate, regard the ordinary potassium salt as an isocyanate. Other chemists think too much stress is laid upon this and allied facts, since frequent observations have shown that normal cyanic compounds readily isomerise. Similarly, allyl thiocyanate readily changes into the isothiocyanate, and the normal cyanuric esters change to the corresponding isocyanuric esters."

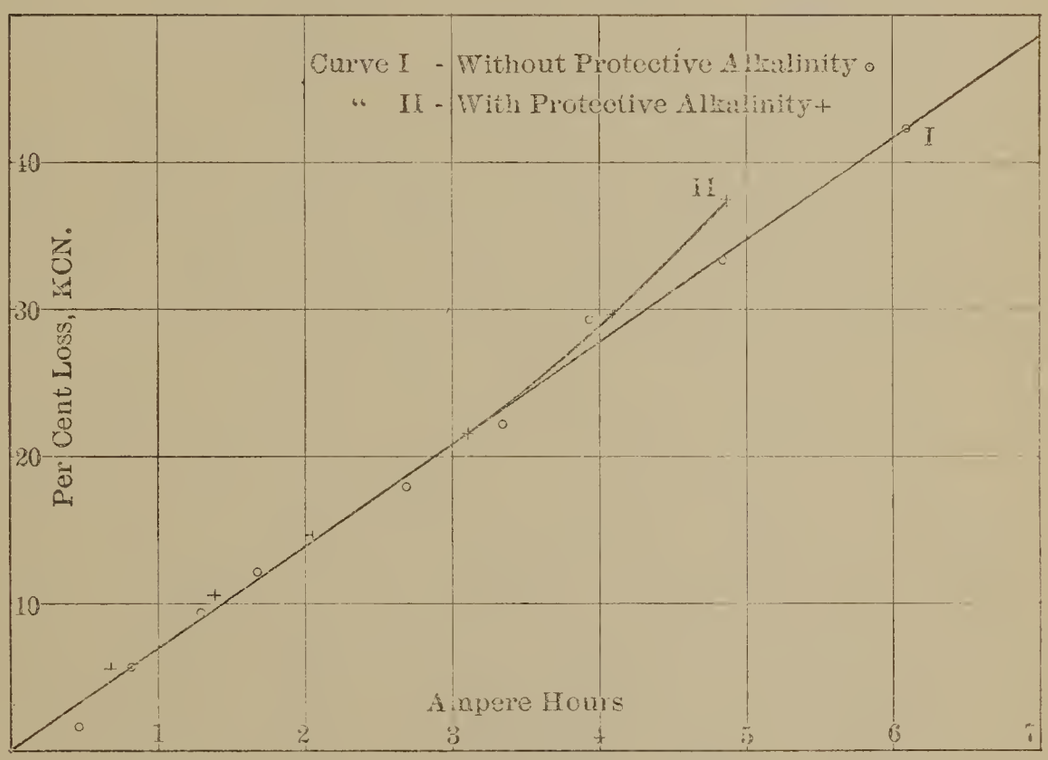

FIG. 1.

Methods of Analysis.

It at once became apparent that one of the chief problems was the finding of satisfactory methods of analysis for the electrolyzed solutions.

Qualitative Test for Cyanates. Cyanates may be detected by the application of Blomstrand's color-reaction, as described by Schneider.[7] A strong solution of the sample is decomposed by passing CO_2 into it until no more HCN is evolved (3 grams of

[6] Jour. Soc. Chem. Ind., p. 887 (1895).

[7] Commercial Organic Analysis, second edition, London, 3 [3], 482 (1896).

KCN are decomposed in forty-five minutes); then sufficient 95 percent alcohol is added to precipitate the K_2CO_3 formed Filter, and add to the filtrate sufficient acetic acid to make the solution slightly acid, and also a small amount of cobalt acetate solution. If cyanates are present, an intense blue color is produced, due to the formation of the double cyanate of cobalt and potassium. As little as 0.35 percent of cyanate is said to be easily detected by this method. We found that this method does not give satisfactory results when used upon weak solutions.

Quantitative Determinations of Cyanates. Feldtmann and Bettel[8] give a method which is said to only serve for the estimation of isocyanates(?). The method is based upon the fact that when $AgNO_3$ is added to a solution containing cyanide, isocyanate, sulphocyanate and ferrocyanide, chloride, carbonate and bicarbonate, the precipitate which is formed consists of silver cyanide, silver isocyanate, silver sulphocyanate, silver ferrocyanide, silver chloride and silver carbonate. Alkaline carbonates are converted into the bicarbonates by the addition of a cold solution of CO_2 in H_2O. The bicarbonates are said not to produce a precipitate with $AgNO_3$. The solution is then titrated with standard $AgNO_3$, using two drops of $KCrO_4$ as an indicator, as in Vielhaber's[9] method for determining KCN. It is necessary to determine the other constituents present which are precipitated by $AgNO_3$ by independent methods, and calculate the total amount of $AgNO_3$ used in precipitating them. This, deducted from the total amount indicated by the titration, gives the amount used in precipitating cyanates. The percentage of cyanate may then be readily calculated. Although we did not investigate this method, we are inclined to question its accuracy.

Herting[10] recommends a method based upon the fact that cyanides are decomposed by mineral acids with the evolution of HCN, while cyanates are decomposed as shown by the below equation:

$$KCNO + 2HCl + H_2O = KCl + NH_4Cl + CO_2$$

The nitrogen in the solution, after treatment with an acid, is determined as NH_3 by distillation with NaOH. The percentage

[8] Proc. Chem. and Met. Soc. of South Africa, **1**, 272.
[9] Arch. Pharm., **13** [3], 408.
[10] Zeitschrift f. Angew. Chem., **24**, p. 585 (1901).

of cyanate is calculated from the amount of nitrogen found. This method was not investigated, as it did not appear to work in well with the determination of the other constituents of the solution.

Victor[11] gives a method for the quantitative estimation of cyanate in the presence of cyanide, based upon the fact that both the cyanides and cyanates are completely precipitated by silver nitrate as AgCN and AgCNO in neutral solutions, while only cyanide is precipitated in a solution slightly acid with HNO_3.

Method. Place two portions of the solution, of 10 cc. each, in 100 cc. flasks and add a known amount of standard $AgNO_3$ solution. (The amount of $AgNO_3$ added should be more than sufficient to precipitate all the salts which are precipitated by this reagent.) One lot is diluted to the mark, filtered, and the excess $AgNO_3$ determined in 50 cc. of the filtrate by Volhard's method, titrating with either a standard solution of KSCN or NH_4SCN and using ferric alum as an indicator. To the other lot add 10 cc. of 1:1 HNO_3, dilute to the mark, filter, and determine the excess $AgNO_3$ in 50 cc. of the filtrate, as with the other portion. The difference between the $AgNO_3$ found in each case is the amount combined with the cyanate. From this the cyanate present is calculated.

In order to test the accuracy of the method a solution of known strength was prepared and analyzed for both KCN and KCNO. The results were not satisfactory, and determinations made using 1 cc. and 5 cc. of nitric acid did not improve them.

Mellor[12] has proposed a method similar to that of Victor. He provides for the removal of carbonates by means of $Ca(NO_3)_2$. He points out that $Ba(NO_3)_2$, as suggested by Victor for this purpose, cannot be used on account of the insolubility of $Ba(CNO)_2$. This method also failed to give satisfactory results. Among other things, the cause for the failure of these two methods appears to be insufficient provision for the removal of carbonates and hydroxides.

Paterno and Pannain,[13] in the course of their work of finding the most favorable conditions for the oxidation of cyanide to

[11] Zeitschrift f. Anal. Chem., 40 [3], 462 (1901).
Jour. Soc. Chem. Ind., 20, p. 1031.

[12] Zeitschrift f. Anal. Chem., 34 [1], p 17 (1901).

[13] Gazetta Chimica Italiana, 34 [2], 152.

cyanate electrolytically, have developed methods of analysis which we found capable of giving satisfactory results for technical work.

The methods are given below in detail:

1. To a 10 cc. sample add a known excess of standard $AgNO_3$ solution. Dilute to 100 cc. and filter. In 50 cc. of the filtrate determine the excess $AgNO_3$ by Volhard's method. The $AgNO_3$ added minus that found in the filtrate gives the amount consumed by the cyanide, cyanate, carbonate and hydroxide. (Note.—This would also indicate the amount consumed by anything else precipitated by $AgNO_3$ in neutral solution.)

2. To a second 10 cc. sample add a known excess of $AgNO_3$ and an excess of acetic acid. Dilute to 100 cc. and filter. In 50 cc. of the filtrate determine the excess $AgNO_3$, as above. The difference between the $AgNO_3$ used in each case is consumed by the precipitation of AgCN and AgCNO.

3. To a third 10 cc. sample add a known excess of $AgNO_3$ and an excess of dilute HNO_3. Dilute to 100 cc. In 50 cc. of the filtrate determine the excess $AgNO_3$, as above. The difference between the $AgNO_3$ consumed in each case is used in the precipitation of the KCN.

The $AgNO_3$ consumed in (2) minus that consumed in (3) is the amount of $AgNO_3$ used in precipitating AgCNO.

4. To a 50 cc. sample add an excess of $Ba(NO_3)_2$. Dilute to 100 cc., filter rapidly, and to 20 cc. of the filtrate, corresponding to a 10 cc. sample of the original solution, add a known excess of $AgNO_3$. Dilute to 100 cc. and determine the excess $AgNO_3$ in 50 cc. of the filtrate. The $AgNO_3$ consumed precipitates AgCN, AgCNO and Ag_2O. Subtract this amount from (1) and there remains the $AgNO_3$ used by the carbonate in solution. Subtract (2) from (4) and the $AgNO_3$ used by the hydroxide is obtained.

In order to test their methods these experimenters made up a solution containing the decinormal equivalent of KCN, KCNO, K_2CO_3 and KOH; 10 cc. therefore contained KCN = 0.00651 g., KCNO = 0.00811 g., K_2CO_3 = 0.00691 g., KOH = 0.00561 g. When analyzed as described above, the results obtained were: (1) Required 40.05 cc. $AgNO_3$ per 10 cc.; (2) required 19.95 cc. $AgNO_3$ per 10 cc.; (3) required 9.95 cc. $AgNO_3$ per 10 cc.;

(4) required 30 cc. of $AgNO_3$ per 10 cc. The $AgNO_3$ used in this case was also decinormal.

The carbonate and hydroxide results were 0.5 percent high, the cyanate correct, and the cyanide 0.5 percent low. Paterno and Pannain state that with varied proportions of the above constituents the same good results were always obtained.

Final Experiments with Alkaline and Neutral Cyanide Solutions.

Three solutions containing about the same amount of KCN were prepared. One contained no protective alkalinity; one contained protective alkalinity due to dissolved $Ca(OH)_2$, and one contained protective alaklinity due to KOH. Each solution was analyzed for KCN, KCNO, K_2CO_3 and KOH. The major portions of each were then electrolyzed and immediately analyzed for the same constituents. Both the electrolyzed and original portions of the solutions were then set aside in separate stoppered bottles and allowed to stand in a dark, cool place for varying periods, as noted in each experiment, when they were again analyzed for each of the above constituents.

Experiment III. A solution was made up containing both KCN and KOH. The KCN, indicated by Liebig's method, was 0.805 percent, and the "protective alkalinity," by Clennell's method, was 85 points. About 1,400 cc. of the above solution was electrolyzed, keeping the ampere reading as near 2 as possible by varying the distance between the electrodes. During electrolysis the temperature rose from 14° C. to 39° C., and the voltage from 6 to 7.5. The total current used was 5 ampere hours.

Analysis of Solution Before Electrolysis. As an example of the method of analysis and the computation of the results the figures upon the solution before electrolysis are given in detail.

Fifty cc. of standard $AgNO_3$ were added in each case. The precipitations were made in 250 cc. flasks, and 100 cc. samples of the filtrate were used for the estimation of the $AgNO_3$ by the Volhard method.

1. Required 1.12 cc. NH_4SCN for excess $AgNO_3$. NH_4SCN

standard 1 cc. = 0.992 cc. $AgNO_3$. 50 — (2.5 × 0.992 × 1.12) = 47.22 cc. $AgNO_3$ used by KCN, KCNO, K_2CO_3 and KOH.

2. Added 10 cc. 1:1 acetic acid. Required 4.69 cc. NH_4SCN. 50 — (2.5 × 0.992 × 4.69) = 38.37 cc. $AgNO_3$ used by KCNO and KCN.

3. Added 10 cc. 1:1 HNO_3. Required 4.76 cc. NH_4SCN. 50 — (2.5 × 0.992 × 4.76) = 38.20 cc. $AgNO_3$ used against KCN.

4. Added excess of $Ba(NO_3)_2$, diluted to 250 cc., took 50 cc. of filtrate, equal to 10 cc. of original. Required 1.76 cc. NH_4SCN for 90 cc., final sample. 50 — (10/9 × 0.992 × 2.5 × 1.76) = 45.15 cc. $AgNO_3$ consumed by KCN, KCNO and KOH.

(*c*)	38.37 — 38.20 =	0.17	cc.	$AgNO_3$	used	by	KCNO.
(*d*)		38.20	"	"	"	"	KCN.
(*a*)	47.22 — 45.15 =	2.07	"	"	"	"	K_2CO_3.
(*b*)	45.15 — 38.37 =	6.78	"	"	"	"	KOH.

(*a*)

$$K_2CO_3 + 2AgNO_3 = AgCO_3 + 2KNO_3$$

138.2 339.78

$$2.07 \times 0.006528 \times \frac{138.2}{339.78} = 0.0055 \text{ g. } K_2CO_3 \text{ per 10 cc.} = 0.055\%$$

(NOTE.—1 cc. $AgNO_3$ solution contains 0.006528 g. $AgNO_3$.)

(*b*)

$$2KOH + 2AgNO_3 = Ag_2O + H_2O + 2KNO_3$$

112.22 339.78

$$6.78 \times 0.006528 \times \frac{112.22}{339.78} = 0.0146 \text{ g. KOH per 10 cc.} = 0.146\%$$

(*c*)

$$KCNO + AgNO_3 = KNO_3 + AgCNO$$

81.11 169.89

$$0.17 \times 0.006528 \times \frac{81.11}{169.89} = 0.0005 \text{ g. KCNO per 10 cc.} = 0.005\%$$

(*d*)

$$KCN + AgNO_3 = AgCN + KNO_3$$

65.11 169.89

$$38.2 \times 0.006528 \times \frac{65.11}{169.89} = 0.0956 \text{ g. KCN per 10 cc.} = 0.956\%$$

It will be noted that the KCN indicated by Liebig's method is low.

Analysis of Solution After Electrolysis.

K_2CO_3	per	10	cc.	0.0161 g.	= 0.161%
KOH	"	"	"	0.0112	= 0.112
KCNO	"	"	"	0.0152	= 0.152
KCN	"	"	"	0.0718	= 0.718

Loss of	KCN	during	electrolysis	per	10	cc.,	0.0238 g.
" "	KOH	"	"	"	"	"	0.0034
Gain "	K_2CO_3	"	"	"	"	"	0.0106
" "	KCNO	"	"	"	"	"	0.0147

Calculated elementary analysis *before* electrolysis:

K, 0.709%; C, 0.182%; N, 0.207%; O, 0.062%; H, 0.003%

Calculated elementary analysis *after* electrolysis:

K, 0.674%; C, 0.169%; N, 0.181%; O, 0.118%; H, 0.002%

Percent of original:

K, 95.0; C, 92.8; N, 87.5; O, 190.5; H, 66.6

In order to determine the effect of standing, both the original and electrolyzed solutions were again analyzed after standing fifty-four days.

Original solution after standing fifty-four days:

	K_2CO_3	KOH	KCNO	KCN	g. per 10 cc.
	0.0065	0.0155	0.0004	0.0938	
Loss (—) or gain (+)	+0.0010	+0.0009	—0.0001	—0.0018	

Electrolyzed solution after standing fifty-four days:

	K_2CO_3	KOH	KCNO	KCN	g. per 10 cc.
	0.0188	0.0119	0.0162	0.0706	
Loss (—) or gain (+)	+0.0027	+0.0007	+0.0010	—0.0012	

Experiment IV. A solution was made up containing both KCN and $Ca(OH)_2$. About 1,400 cc. of this solution was electrolyzed under the same conditions which obtained in Experiment III, *i. e.*, ampere reading kept at 2 throughout the experiment. The temperature rose from 14° C. to 28° C., and the voltage varied from 6.5 to 7. It will be noted that the temperature rise is not so great as before. The total current used was 5 ampere hours.

Analysis of Solution Before Electrolysis.

$CaCO_3$	None in filtered solution				
$Ca(OH)_2$	per	10	cc.	0.0139 g.	= 0.139%
KCNO	"	"	"	0.0008	= 0.008
KCN	"	"	"	0.0898	= 0.898

Analysis of Solution After Electrolysis.

$CaCO_3$	per	10	cc.	0.0130 g.	= 0.130%
$Ca(OH)_2$	"	"	"	0.0043	= 0.043
KOH	"	"	"	0.0079	= 0.079
KCNO	"	"	"	0.0080	= 0.080
KCN	"	"	"	0.0670	= 0.670

NOTE.—$CaCO_3$ was determined by thoroughly mixing the electrolyte by shaking, and then taking two portions of 100 c.c. each, containing the $CaCO_3$ in suspension. These were filtered, ignited to CaO and weighed. The CaO found, was calculated to $CaCO_3$.

Loss of	$Ca(OH)_2$	during	electrolysis	per	10	cc.,	0.0096 g.
" "	KCN	"	"	"	"	"	0.0228
Gain "	KOH	"	"	"	"	"	0.0079
" "	$CaCO_3$	"	"	"	"	"	0.0130
" "	KCNO	"	"	"	"	"	0.0072

Calculated elementary analysis *before* electrolysis:

K, 0.539%; C, 0.166%; N, 0.194%; O, 0.060%; Ca, 0.075%; H, 0.004%

Calculated elementary analysis *after* electrolysis:

K, 0.496%; C, 0.151%; N, 0.158%; O, 0.119%; Ca, 0.075%; H, 0.003%

Percent of original:

K, 92.0; C, 91.0; N. 81.5; O, 198.4; Ca, 100.0; H, 75.0

Both the original and electrolyzed solutions were again analyzed after standing fifty-one days in stoppered bottles.

Original solution after standing fifty-one days:

	KCNO	KCN	g. per 10 cc.
	0.0005	0.0875	
Loss (—) or gain (+)	—0.0003	—0.0023	

Electrolyzed solution after standing fifty-one days:

	KCNO	KCN	g. per 10 cc.
	0.0140	0.0672	
Loss (—) or gain (+)	+0.006	+0.0002	

Experiment V. About 1,400 cc. of KCN solution containing no protective alkalinity was made up and electrolyzed under the

same conditions as obtained in Experiments III and IV, *i. e.*, ampere reading kept at 2 throughout the experiment. Voltage 5.5 to 7. The total current used was 5 ampere hours.

Analysis of Solution Before Electrolysis.

K_2CO_3	per 10 cc.	0.0046 g.	= 0.046%
KOH	" " "	0.0000	= 0.000
KCNO	" " "	0.0000	= 0.000
KCN	" " "	0.0950	= 0.950

Analysis of Solution After Electrolysis.

K_2CO_3	per 10 cc.	0.0023 g.	= 0.023%
KOH	" " "	0.0063	= 0.063
KCNO	" " "	0.0070	= 0.070
KCN	" " "	0.0738	= 0.738

Loss of	K_2CO_3	during electrolysis	per 10 cc.	0.0023 g.
" "	KCN	" "	" " "	0.0212
Gain "	KOH	" "	" " "	0.0063
" "	KCNO	" "	" " "	0.0070

Calculated elementary analysis *before* electrolysis:

K, 0.596%; C, 0.179%; N, 0.204%; O, 0.016%; H, 0.0%

Calculated elementary analysis *after* electrolysis:

K, 0.534%; C, 0.148%; N, 0.171%; O, 0.040%; H, 0.001%

Percent of original:

K, 89.6; C, 82.9; N, 83.9; O, 240.0

Both the original and electrolyzed solutions were again analyzed after standing forty-four days in stoppered bottles.

Original solution after standing forty-four days:

	K_2CO_3	KOH	KCNO	KCN	g. per 10 cc.
	0.0025	0.0001	0.0002	0.0945	
Loss (—) or gain (+)	—0.0021	+0.0001	+0.0002	—0.0005	

Electrolyzed solution after standing forty-four days:

	K_2CO_3	KOH	KCNO	KCN	g. per 10 cc.
	0.0097	0.0031	0.0084	0.0724	
Loss (—) or gain (+)	+0.0074	—0.0032	+0.0014	—0.0014	

Table III.

Resumé of the Results of Experiments III, IV and V.

Treatment	KCN Percent	KCNO Percent	KOH Percent	$Ca(OH)_2$ Percent	K_2CO_3 Percent	$CaCO_3$ Percent
Experiment V. KCN						
Before Electrolysis . . .	0.950	0.000	0.000	. . .	0.046	. . .
After standing 44 days	0.945	0.002	0.001	. . .	0.025	. . .
Gain +, Loss — . . .	—0.005	+0.002	+0.001	. . .	—0.021	. . .
After Electrolysis . . .	0.738	0.070	0.063	. .	0.023	. . .
After standing 44 days	0.724	0.084	0.031	. . .	0.097	. . .
Gain +, Loss — . . .	—0.014	+0.014	—0.032	. . .	+0.074	. . .
Experiment III. KCN + KOH						
Before Electrolysis . . .	0.956	0.005	0.146	. . .	0.055	. . .
After standing 54 days	0.938	0.004	0.155	. . .	0.065	. . .
Gain +, Loss — . . .	—0.018	—0.001	+0.009	. . .	+0.010	. . .
After Elecrolysis . . .	0.718	0.152	0.112	. . .	0.161	. . .
After standing 54 days	0.706	0.162	0.119	. . .	0.188	. . .
Gain +, Loss — . . .	—0.012	+0.010	+0.007	. . .	+0.027	. . .
Experiment IV. KCN + $Ca(OH)_2$						
Before Electrolysis . . .	0.898	0.008	0.000	0.139	0.000	0.000
After standing 51 days	0.875	0.005	. . .	. . .	. . .	. . .
Gain +, Loss — . . .	—0.023	—0.003	. . .	. . .	. . .	. . .
After Electrolysis . . .	0.670	0.080	0.079	0.043	0.000	0.130
After standing 51 days	0.672	0.140	. . .	. . .	. . .	. . .
Gain +, Loss — . . .	+0.002	+0.060	. . .	. . .	. . .	. .

Note.—The cyanate and carbonate indicated in the original solutions may have been due to the fact that some of the solutions stood for several days before being analyzed.

Reactions Occurring During and After Electrolysis.

(1) $KCN + \text{electrolysis} = K + CN.$

(2) $H_2O + \text{electrolysis} = H_2 + O.$

The K liberated at the cathode combines with water to form KOH and H, as shown by equation (3).

(3) $K + H_2O = KOH + H.$

CN formed at the anode may combine directly with H formed at the cathode to form HCN, as shown by equation (4).

(4) $CN + H = HCN.$

HCN thus formed may react with KOH present to again form KCN, as shown by equation (5).

(5) $KOH + HCN = KCN + H_2O$.

HCN might react with K_2CO_3, a product of the decomposition of KCNO, to form KCN, H_2O and CO_2, as shown by equation (6).

(6) $K_2CO_3 + 2HCN = 2KCN + H_2O + CO_2$.

HCN with nascent H may form NH_2CH_3 (methylamine), according to equation (7).

(7) $HCN + 2H_2 = NH_2CH_3$.

HCN, upon standing in the presence of an alkali or acid. breaks up, forming NH_3 and H_2CO_2 (formic acid). In the presence of an alkali the formate of the alkali is formed. The reactions involved are shown by equations (8), (9) and (10).

(8) $HCN + 2H_2O = H_2CO_2 + NH_3$.

(9) $2KOH + H_2CO_2 = K_2CO_2 + 2H_2O$.

(10) $Ca(OH)_2 + H_2CO_2 = CaCO_2 + 2H_2O$.

O liberated at the anode might combine directly with KCN to form KCNO, according to equation (11).

(11) $2KCN + O_2 = 2KCNO$.

CN liberated at the anode reacts with KOH to form KCN, KCNO and H_2O. This is, perhaps, by far the most important reaction for the formation of cyanate, and is also the most important primary reaction taking place in cyanide solutions containing free alkali, when they are electrolyzed. This reaction is shown by equation (12).

Paterno and Pannain[14] have shown that the formation of KCNO in a solution of KCN containing KOH under favorable conditions may amount to 92.8 percent of the KCN decomposed by the current. KCNO is therefore the principal product formed in solutions of KCN containing KOH.

(12) $2KOH + 2CN = KCN + KCNO + H_2O$.

Masson and Masson[15] have investigated the decomposition of metallic cyanates from the biuretic standpoint. They divide them

[14] Gazetta Chimica Italiana, **34** [2], 152.
[15] Zeitschrift f. Physick. Chem., **70**, p. 290.

into two classes: (*a*) Cyanates of metals forming insoluble carbonates; (*b*) cyanates of metals forming soluble carbonates. They assumed as a working hypothesis that the reaction proceeded as two successive ones, first a slow unimolecular reaction of the cyanic ion with H_2O, followed by a relatively rapid reaction in which CNO^- and NH_4^+ are converted into urea. They represent the reaction of a cyanate with a base forming an insoluble carbonate by the following equations:

$$(13)\quad CNO^- + 2H_2O \rightarrow NH_4CO_3$$
$$Ca^+ + NH_4CO_3 \rightarrow NH_4^+ + CaCO_3.$$

$$(14)\quad CNO^- + NH_4^+ \rightarrow CO(NH_2)_2.$$

NaCNO and KCNO, which belong to the second class of cyanates, both behave the same, but differently from the first class of cyanates just considered. The following equation represents the reaction which takes place:

$$(15)\quad 4KCNO + 6H_2O = 2K_2CO_3 + (NH_4)_2CO_3 + CO(NH_2)_2.$$

It appears possible that KCNO could be reduced by nascent H forming NH_3, $KHCO_3$ and KCN, as shown by equation (16).

$$(16)\quad 3KCNO + 4H = NH_3 + KHCO_3 + 2KCN.$$

According to Hamilton,[16] KCNO might react with H_2O to form $KHCO_3$ and NH_3, as shown by equation (17).

$$(17)\quad KCNO + 2H_2O = KHCO_3 + NH_3.$$

$KHCO_3$ with KOH forms K_2CO_3 and H_2O, as shown by equation (18).

$$(18)\quad KHCO_3 + KOH = K_2CO_3 + H_2O.$$

Hamilton[16] also gives the following equation:

$$(19)\quad KHCO_3 + Ca(OH)_2 = CaCO_3 + KOH + H_2O.$$

Inspection of the calculated elementary analyses, before and after electrolysis, shows a loss of all elements except O, of which there is a marked gain. The source of the O is through the decomposition of water.

The K shows the smallest loss in the solution containing KOH and the greatest loss in the solution containing no protective alkalinity. The solution containing $Ca(OH)_2$ shows a loss inter-

[16] Proc. Chem. and Met. Soc. of S. A., 4, p. 357.

mediate between the other two. This apparent loss of K is doubtless due to the formation of K salts not precipitated by $AgNO_3$. In the one experiment in which Ca salts were present there was apparently no loss of the Ca.

The loss of the C, although in each case somewhat greater than the K, is the least with the solution containing KOH and the greatest with the solution containing no alkalinity. The loss of C is probably largely due to the formation of gases which escape from the solution CO_2, CN and HCN. As might be expected, the loss of gases containing carbon is the greatest in the solution containing no protective alkalinity.

The O shows a large gain in all the solutions, but appears to be the greatest in the solution containing no protective alkalinity.

The H shows a loss in both the solutions containing protective alkalinity and only a slight gain in the solution containing no protective alkalinity. Although a large amount of H results from the decomposition of water, it will be noted that its behavior is quite different from that of the O.

The loss of N is the greatest in the solution containing Ca and the least in the solution containing KOH. The loss of N may be, at least in part, accounted for by the formation of NH_3, CN and HCN.

With the KCN solution containing no alkali the loss of KCN amounted to 22.3 percent of that started with, but the formation of KCNO is less than half of that with the solution containing KOH. There is a loss of K_2CO_3 and a small gain in KOH. A considerable part of the loss of cyanide under these conditions must be due to the formation of gases or compounds not precipitated by $AgNO_3$, possibly largely the former. Upon standing, there is a loss of both KCN and KOH, a small gain in KCNO and a marked gain in K_2CO_3.

With the KCN solution containing KOH the loss of KCN amounted to 25 percent of that started with. The chief effect appears to be the formation of KCNO and K_2CO_3 and the loss of KOH. Upon standing, there is a marked gain in both the KCNO and K_2CO_3, but especially of the K_2CO_3. There is a slight gain in the KOH and the usual loss of KCN upon standing.

With the KCN solution containing $Ca(OH)_2$ the loss of KCN

amounted to 25.39 percent of that started with. The KCNO in solution after electrolysis is not much greater than when no alkali is present. There is a loss of the $Ca(OH)_2$ through the precipitation of $CaCO_3$. There is a marked gain in KOH.

The principal effect appears to be the formation of $CaCO_3$. Upon standing, this solution showed a most marked gain in KCNO. The KCN showed a very slight gain.

The decomposition of the fresh solution, upon standing, appears to be largely due to contact with air, for the reason that when the bottles were completely filled with solution there was little or no decomposition.

CONCLUSIONS.

1. The bulk of the decomposition of cyanide occurring during electrolysis is due to oxygen liberated at the anode through the decomposition of water.

2. The principal final reaction in solutions of cyanide containing protective alkalinity, which in cyanide mill practice is usually due to lime, is the formation of the corresponding alkali carbonate. This accounts for the formation of large amounts of calcium carbonate during the electrolytic precipitation of gold and silver from cyanide solutions.

3. A consideration of the reactions previously discussed will explain the failure in many cases to obtain regeneration of cyanide when such regeneration is theoretically possible. It is apparent that the decomposition of cyanide may easily be equal to or greater than that regenerated through the precipitation of the metal in solution.

4. The Liebig titration for the estimation of cyanide in an electrolyzed solution gives results which are low.

5. The changes taking place in fresh and electrolyzed cyanide solutions, upon standing, are different.

6. While not wishing to discourage investigators working along the lines of aiding extraction with refractory ores by electrolysis, we think that it is of importance to call attention to the disadvantages which may arise through the indiscriminate use of electrolysis with an ore pulp. First, a large loss of cyanide

may result from which there is no benefit derived, and, second, electrolysis may actually interfere with extraction through the formation of a coating of calcium carbonate upon the ore particles.

DISCUSSION.

F. A. LIDBURY: I have been very much interested in both the papers by Prof. Kern and Mr. Clevenger on the subject of the electrolysis of alkaline cyanides, but the objects of the investigations have not been quite clear to me. I gather, however, that the use of electrolysis in connection with these solutions in practice may have the following objects:

The electrolytic precipitation of gold and silver from cyanide solutions; that is perfectly clear.

The regeneration of cyanide when such regeneration is theoretically possible; that is also fairly clear.

For aiding the extraction by electrolysis; that is not at all clear to me unless it is based on a misunderstood conception of electrolysis, which leads many people to think that it is a kind of specific for all sorts of bad metallurgical digestion, and that the mere insertion of an anode or a cathode into any old thing will produce wonderful results obtainable by no other means. I do not know whether I am right in so reading the words in conclusion No. 6, but the point which Mr. Clevenger's paper was directed against seems to have been the idea that all that was necessary was to go ahead and electrolyze something or other to get wonderful results; if that is so, it appears to me to be slaughtering a very, very dead dog.

In considering any application of electrolytic methods, it should be borne in mind, as Prof. Bancroft has been preaching for years and years, that all electrolytic effects are essentially chemical in their nature, and that if you do not understand the chemistry of the problem it is of no use to lug in electrolysis to help your understanding. One thing that has struck me most particularly in these two papers is the absence of any very clear idea of what electrolysis is to be called upon to do. Unless we understand in the first case what compounds beside cyanide assist

extractions, it is rather ludicrous to expect to get these unknown compounds by electrolyzing cyanides under any old sort of conditions that happen to come handy.

The first thing to be solved in any general investigation of this kind is: What are the actual compounds or the actual reactions that we desire to produce? Say, for instance, that one of the objects of cyanide solutions is the regeneration of cyanides from cyanates, etc. One would not consider that by any means an impossible problem; at any rate, it would be one worth a very much more careful and thorough investigation than is presented to us in either of the papers on this subject. Mr. Clevenger has somewhat shielded himself from the full force of my wrath by making a belated apology for not having investigated the electrolysis in the proper manner. I was going to point out very strongly what he does not mention at all in his paper, but what he casually alluded to in his abstract, that the only way to investigate an electrolytic problem of this nature is to investigate phenomena at the anode and cathode separately. As far as I know, that has not been attempted, and until it is we will have no light on the subject, since it is perfectly ridiculous to expect that cyanide can be regenerated by putting in a couple of electrodes and letting them whiz.

Not only is it necessary to investigate the products of reaction at the anode and cathode separately, but it is also necessary to conduct such an investigation with very strict observance of constancy of conditions of any kind which might eventually affect the result, such as the maintenance, through any one particular experiment, of a certain temperature, and of a certain potential drop between the anode or cathode and the solution; in neither of these investigations has any attention been paid to such points, and in Mr. Clevenger's paper he states that gases are evolved, but makes a guess at these gases. In electrolysis there is nothing that deserves more careful investigation or gives more abundant, rapid and reliable information than the examination of evolved gases.

I think I have said enough to show that if these questions were investigated in the proper manner we would have some idea of what takes place at the anode and cathode and not a general jumble when both reactions are intermixed, and there

would be no need of printing a lot of hypothetical reactions, most of which are probably, and some certainly, inaccurate.

G. H. CLEVENGER: Mr. Lidbury has stated in a very concise manner, and perhaps more strongly than I have, the conclusion No. 6. On the other hand, he must bear in mind that this paper is not final by any means, and, further than that, the electrolyses in this case were carried on under just such conditions as various men have worked under in the plants all over the country, with the idea of showing what happens under these particular conditions. My principal mission in this paper has been to call the attention of Western metallurgists to this point, and, of course, the valuable discussion contributed by Mr. Lidbury will show the lines along which to proceed in prosecuting further work. Those of us of the West would be very glad if Mr. Lidbury or anyone else who may have the proper ideas of electrolysis would take up this problem and work it out in a thorough and comprehensive manner. We need it.

F. C. FRARY: There are some of these reactions that do not look right. I wanted to call particular attention to them because in some cases a person might very easily misunderstand the process involved.

The sixth reaction is the opposite of what we know does actually take place. It is very well known that carbon dioxide reacts with potassium cyanide, giving off free hydrocyanic acid, and that potassium cyanide is so far hydrolyzed in aqueous solution as to contain considerable free hydrocyanic acid. There would undoubtedly be an equilibrium set up between the two acids and their salts, but it hardly seems legitimate to give the reaction in the form in which it appears here.

The thirteenth reaction likewise represents impossible things as it stands, involving what appears to be ammonium percarbonate, but I judge that it is a misprint or a mistake in copying from the original sources, and that all its members except the water and calcium carbonate should be represented as ions, not molecules. If so, it represents merely a more difficult form of equation seventeen.

The reason for the fifteenth reaction is not clear to me. I do not see why such a complicated method of formation of urea

and carbonate together must be assumed instead of the simple transformation of ammonium cyanate into urea (Equation 14).

G. H. CLEVENGER: I might say in regard to the reactions which Mr. Frary has just criticized, more particularly 13, 14 and 15, that these are not reactions for which I am responsible, but reactions which have been found in looking up the literature upon the subject of the decomposition of cyanates, and, of course, that is true of a great many of the other reactions. In fact, the bulk of them are reactions which others have suggested, and in some cases, perhaps, I have fallen a little bit into error in not having given the direct reference to each reaction; but for these last reactions, which have been criticized, I have given the reference and have introduced them at this point for the sake of discussion.

F. A. LIDBURY: I hope I may be pardoned for adding one word to the remarks that Mr. Clevenger replied to. He rather missed the main point of these remarks, which possibly I did not lay as much stress on as I did on the methods of electrolytic investigation. What I meant to say was that it is of no earthly use trying to begin an investigation on the results of electrolysis until you know what the chemical results you want are. When you come, for instance, to the object of assisting the dissolution of metals from refractory ores, you must know what the compounds are that would assist such extractions. On that point in particular there appears to be a lack of clear thinking and clear statement, and that is the point I wish to emphasize.

J. W. RICHARDS: I think the reason some of these things are not stated clearly is that they are well known to metallurgists who are familiar with the cyanide process, and Mr. Clevenger has taken a knowledge of them for granted; for instance, take this sixth conclusion, which Mr. Lidbury says is indistinctly stated. The statement there alludes to the fact that many metallurgists have had the idea of treating an ore pulp with cyanide solution and putting in electrodes so as to simultaneously throw down the gold, so that in one operation the gold is dissolved and precipitated electrically. There have been a good many methods and ideas of working out that process, and Mr. Clevenger alludes to it in a brief way. It was not the object of his paper, but

his remarks under that head are quite understandable, and I think his contentions are correct.

As to the other things that take place in the solution, it must be remembered that in a cyanide solution the metals are present in an extremely small proportion, and when you pass the current through you can hardly get more than 2 percent current efficiency; the great problem of the electrolytic precipitation is to render the 98 percent of current which does not deposit any metal harmless as far as the cyanide is concerned, that is, to prevent it from oxidizing the cyanide, forming cyanate or liberating hydrocyanic acid, or combining with it and producing insoluble compounds. It is the consideration of that fact, that only a small proportion of the current can be utilized and the other and larger part has to be taken care of and rendered harmless, which is here the hard problem of the electrometallurgist.

I do not share Mr. Lidbury's sweeping statement as to the reactions; the most of those, with one or two exceptions which have been pointed out by Mr. Frary, are understandable and clearly stated, and state the problem in a fair way, and Mr. Clevenger's position in showing what happens in the ordinary electrolysis of cyanide solutions is understandable also, because he wants to show what is taking place under practical conditions. It is almost impracticable to separate the anode and cathode solutions in ordinary practice, and therefore the study of what takes place under the ordinary conditions is a very proper object This, however, can certainly be supplemented, and more light thrown on it, by further investigation in the laboratory under conditions differing from those in practice.

Mr. Clevenger stated yesterday that the lead peroxide anodes used as insoluble anodes were not permanent, but would last about a year in practice, and therefore were very far from being satisfactory. I have been credibly informed by a member of the Society who has worked a great deal with insoluble lead anodes that, if these anodes are peroxidized in a strong alkaline solution before being put into the acid solution, they are more permanent than if oxidized in an acid solution.

G. H. Clevenger: I think a great many of Mr. Lidbury's ideas are splendid, and it would be a great benefit to the cyanide

industry if his expenses could be paid and he be permitted to go around and lecture to these people who are experimenting upon ore treatments. (Laughter.) This is not said in a jocular way. He has the proper idea along what lines further investigation should be carried, that is, to distinctly separate the anode and cathode reactions and to make a very definite study of the subject. There is one point, however, he mentioned in that connection which I think he would not have said so much about if he had been more conversant with cyanide mill conditions, and that is that these tests should be made under rigorous conditions as regards temperature. In the cyanide plants located in the West, where we have considerable changes of temperature, it is not feasible to maintain working solutions at a certain fixed temperature; that is a point which is, no doubt, all right in the laboratory, yet in mill work it is a point not feasible to maintain. So it is with a great many points which may appear to be desirable. A man who is operating a plant always has before him the expense of the operation which he carries on, and very often he has to deliberately sacrifice rigorous scientific treatment for a method which gives him an approximate result and a method which yields the greatest returns in dollars and cents.

In conclusion I want to say that I do not think anyone could have stated this problem more clearly than Mr. Lidbury has regarding future work, particularly with refractory ores, and, if for no other reason, I think my paper has been justified in bringing out that clear statement.

F. A. Lidbury: If Professor Richards' opinion is that the proper way to tackle electrolytic investigations of this nature is to do so by jumbling the whole thing together instead of separating the phenomena at the two electrodes, then all I have to say is that such an attempt would be considered the rankest kind of bad practice in any decent technical laboratory.

In regard to the maintenance of fixed conditions, Mr. Clevenger must have misunderstood my point. Unless you carry out your investigation by conducting such experiment at one particular temperature, under one particular current density, etc., you cannot possibly tell the specific effect of change of any condition on your results. It is all very well to say that you may be working under temperatures not economically obtainable

in practice, but unless you get this information you are not in a position to lay your finger upon the proper economic conditions for practice.

I am obliged to Mr. Clevenger for his suggestion that I should go out and lecture to the people experimenting in ore treatment, but I think I shall be doing more useful work by continuing to lecture to those who do lecture to them.

E. P. SCHOCH: Mr. Lidbury's suggestion that the two electrodes be studied separately leads me to call attention to the manner in which the electrolytic cell and the battery are now brought before beginners in elementary classes in physics and chemistry. The cells are presented as (single) units, which is absolutely wrong, because they are made up of *three* units—the two poles, each with its surrounding liquid, and the liquid conductor between them. The kind of an action taking place in any one is not in the least dependent upon the kind of an action taking place in any one of the others. This should be shown experimentally when such cells are first presented. Then the study of each unit should be taken up separately. The action of any one (whole) cell is then easily understood, because all parts appear in their proper relation, and the action of the whole cell appears as the sum of the actions of its parts. The method of presentation now employed is generally illogical, and electrochemists should correct this error in elementary teaching.

F. W. SKIRROW: It seems to me, whatever standpoint one takes, whether that of a theoretical man or practical man, that no investigation of any character is of much value unless the effect of varying conditions are studied by varying one condition at a time. To carry out an investigation where several conditions vary simultaneously would lead one to results which would not carry much weight in studying the problem. I am speaking more or less as a practical man; I have been in practical work for ten years, and I have in my experience of investigation found that the absolute separation of various factors which were affecting the case was the only way to arrive at a conclusion of a practical nature.

It does not follow if you study a certain thing at a definite temperature that you are going to spend a large amount of

money to keep the plant temperature at that point, but, at any rate, you know what is happening under various conditions. If you have studied conditions one at a time, then when anything goes wrong you are more likely to be able to estimate how far your trouble is due to each of the various factors involved in the case. If you study the problem by varying conditions many at a time, you have no definite information upon which to act, and have not the data upon which to base a suitable compromise.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

SIMULTANEOUS DETERMINATION OF COPPER AND LEAD, WITH THE ROTATING ANODE.

By Arthur J. White.

In electrolysing nitric acid solutions of copper it has been observed that lead tends to hold up some of the copper. More nitric acid is necessary for lead than will permit of copper coming down completely.

Stansbie has shown (Proceedings of Faraday Soc., Nov. 26, '12) that rotation causes copper to be comparatively insoluble in nitric acid, and he recommeds continuing rotation while washing out. He claims that sulphuric acid assists in the deposition, probably because of the formation of a nitrosyl acid instead of nitrous acid, which is the active agent for copper. However, this relative insolubility may be assigned to the formation of ammonium sulphate, no ammonium salt being formed in the absence of sulphuric acid. According to Easton (J. Amer. Chem. Soc., **25**, 1042), nitric acid is quantitatively converted to ammonium sulphate in the presence of an excess of copper sulphate by the current.

The question arose as to whether sulphuric acid would allow of copper being completely precipitated from a nitric acid solution while lead is being determined simultaneously on the anode. It was found that it would, provided that the proportion of lead is not high enough to precipitate lead sulphate. This salt does not appear unless the proportion of lead to copper is greater than one to four, as the copper nitrate exerts a solvent action on smaller quantities.

When less than 0.15 gram of lead is present with 0.60 gram of copper, they are quantitatively precipitated in thirty minutes by a current of 8 amperes at 6 volts.

The best proportions of acid are 4 cc. concentrated nitric acid and 10 drops of concentrated sulphuric acid in a volume of 70

to 80 cc. The anode is rotated at from 400 to 700 R.P.M., and the copper is deposited on a 60-gram platinum dish at $N.D._{100}$.

THE ANODE.

The anode is of a simple form, being a small dish (3.8 cm. diameter) of platinum mounted on the end of a platinum rod 10 cm. long and 0.15 cm. diameter. The entire weight is below 15 grams, and the surface 20 sq. cm.

The surface must be roughened. This is best effected by platinizing from a 5 percent sulphuric acid solution. Several drops of platinum solution corresponding to 0.05 gram metal were used. A current of 0.7 amp. at 1.5 volts for 30 minutes while rotating gave a dense black deposit. On ignition this turned white, and showed no tendency to loosen on continued use.

A larger, more complicated dish anode has been used before in the determination of copper (Languess, J. Am. Chem. Soc., **29**, 459). However, lead was not deposited upon this anode, the surface being smooth.

The ability of the rotating dish anode to hold the lead dioxide was tested.

A solution of lead was prepared containing 0.4990 gram of the pure metal. This was placed in a 30-gram platinum dish at a volume of 40 cc., 5 cc. of free nitric acid being present. The anode was rotated at 1,000 R.P.M., which was higher than was ever allowed for practical work. To the cold solution a current of 8 amperes at 6 volts was applied for 25 minutes. No tendency to splash was noticed on the cover glasses, although the temperature rose to the boiling point. A higher current could not be applied without causing the liquid to boil. After 20 minutes the dish was washed out with 500 cc. distilled water, the rotation being continued throughout. The current dropped to zero, the dish was removed, and, after the water was shaken well from the lead by a minute's rotation, the anode was dried for 15 minutes at a temperature of 130° C. The anodes were always dried with the end of the rod stuck in a large flat cork. The lead should not be touched before weighing. The result was satisfactory. The cathode increased in weight slightly, although the current was stopped for five seconds early in the electrolysis.

PURE LEAD SAMPLE.

Lead Present, Grams	Found		Lead Deposited on Cathode	Error Milligrams	Time Minutes	Speed R. P. M.
	PbO_2	Pb (Factor 0.866)				
0.4990	0.5730	0.4962	0.0021	— 0.7	25	1000

The factor for lead in the dioxide deposit varies considerably with the conditions. Smith recommends the factor 0.8643 when using a sand-blasted platinum dish as anode and rotating the cathode. In this work the theoretical factor 0.866 comes nearer the real condition, and has been used throughout. The temperature of 130° to 135° is sufficient to dry the deposits in 15 minutes. Alcohol and ether were never used previously to drying the lead, although they were always applied to the copper deposits.

PURE LEAD AND COPPER.

A series of experiments was made with varying proportions of pure lead and copper.

The speed was kept between 400 and 700 R.P.M.

$N.D._{100} = 8$ amperes at 6 volts for the cathode, while the anode, with surface of only 20 sq. cm., had a higher density $N.D._{100} = 40$.

One cc. of 25 percent sulphuric acid and nitric acid (sp. gr. 1.42) varying from 3 to 6 cc. were added to the contents of the dish before passing the current.

	Weight of Lead Dioxide			Copper		
	Present	Found	Error Milligrams	Present	Found	Error Milligrams
A	0.0107	0.0104	—0.3	0.6530	0.6526	— 0.4
B	0.0375	0.0374	—0.1	0.6530	0.6530	0.0
C	0.1072	0.1064	—0.8	0.6530	0.6540	+ 1.0
D	0.1072	0.1070	—0.2	0.6530	0.6535	+ 0.5

APPLICATION TO BRASS ANALYSIS.

(a) Preliminary Treatment. Five grams of brass drillings free of oil, are weighed into a tall 1-liter beaker. Ten cc. of

water and 20 cc. strong nitric acid are introduced. All the metal is dissolved in from 5 to 15 minutes. About 150 cc. of hot water is added, and the whole boiled briskly for 5 minutes to expel nitrous fumes. Much turbidity indicates more than a trace of tin, and the meta-stannic acid is filtered off, ignited and weighed. Ordinarily mere traces of tin are present, and the solution is made up to exactly 250 cc., and 50 cc. withdrawn for electrolysis. This sample corresponds to 1.000 gram of brass, and the weight of copper will correspond to percent directly.

(b) Electrolysis. The platinum dish should have a capacity of at least 100 cc., but it need not weigh more than 40 grams. The dish anode is advantageous, but a spiral wire anode will do if the surface has been platinized. Gauze has not been tried under these conditions, but a flat, circular gauze anode would be perfectly applicable. A spiral anode having 5 sq. cm. submerged surface will hold 0.0900 gram of lead dioxide. This limit is seldom exceeded, the average lead percentage being below 4 percent, or corresponding to 0.05 gram of dioxide.

To the 50 cc. sample 4 cc. of strong nitric acid and 1 cc. of 25 percent sulphuric acid were added, and the current of 8 amperes at 6 volts applied for 30 minutes. The cover glasses were generally washed after about 7 minutes, when the blue color had faded from the solution. The washing was twice repeated, and the siphoning was conducted as recommended in "Electro-Analysis," by E. F. Smith.

At the time of washing, the anode must not be nearer the dish than ½ inch, hence the dish is lowered at some time during the latter part of the electrolysis. Rotation is not stopped until the water has been shaken off by rotation in the air. The deposit is dried at 130° C. for 15 minutes. Constant weight is reached in this time, as is shown by further drying and re-weighing.

In case a gray spot of zinc forms on the copper, due to not keeping the anode distant ½ inch while washing, a little hydrochloric acid may be used without impairing the copper deposit appreciably.

Iron is determined in the siphonate by a volumetric method, while the zinc in routine work is taken by difference.

BRASS.

Preliminary Analyses of Pure Metal Mixtures.

	Zinc Present	Weight of PbO_2			Copper		
		Present	Found	Error Milligrams	Present	Found	Error Milligrams
1	0.340	0.1072	0.1070	— 0.2	0.6530	0.6526	— 0.4
2	0.125	0.0574	0.0582	+ 0.8	0.3270	0.3273	+ 0.3
3 (*a*)	0.340	0.0537	0.0543	+ 0.6	0.6500	0.6497	— 0.3
3 (*b*)	0.340	0.0537	0.0540	+ 0.3	0.6500	0.6490	— 1.0
4 (*a*)	0.250	0.1147	0.1145	— 0.2	0.6530	0.6520	— 1.0
4 (*b*)	0.250	0.1147	0.1148	+ 0.1	0.6530	0.6517	— 1.3
5 (*a*)	0.300	0.1730	0.1725	— 0.5	0.5630	0.5628	— 0.2
5 (*b*)	0.300	0.1730	0.1722	— 0.8	0.5630	0.5623	— 0.7

Foundry Brass Samples.

	Zinc Present	Weight of PbO_2			Copper		
		Old Method	Found	Error Milligrams	Present	Found	Error Milligrams
6 (*a*)	0.320	0.0330	0.0322	— 0.8	0.6550	0.6550	0.0
6 (*b*)	0.320	0.0330	0.0329	— 0.1	0.6550	0.6544	— 0.6
7 (*a*)	0.320	0.0340	0.0340	0.0	0.6530	0.6530	0.0
7 (*b*)	0.320	0.0340	0.0339	— 0.1	0.6530	0.6520	— 1.0
7 (*c*)	0.320	0.0340	0.0331	— 0.9	0.6530	0.6535	+ 0.5

Many analyses of brass have been run, but few results have been obtained using both methods on the same metal. After working with the method, and testing the siphonate with hydrogen sulphide, sufficient confidence was gained for its adoption, and the old process of electrolysing for copper and lead separately was abandoned.

Analysis when lead is in comparatively large proportions, i. e., any amount more than ¼ that of the copper. The nitrate solution should contain no unnecessary free acid. Sulphuric acid is added in excess of the lead present. After the lead sulphate has settled, the solution may be decanted off, and more nitric acid added immediately to prevent further precipitation of lead sulphate. The bulk of the lead in the form of sulphate after washing several times with water by decantation is free of copper, and may be dissolved in strong HNO_3 and electrolyzed inde-

pendently. The copper solution is electrolyzed as previously described, enough nitric acid being added to give a concentration of 5 to 6 percent. The necessity for evaporation of the lead siphonate for copper is thus avoided.

EFFECT OF OTHER METALS.

(a) Zinc and Cadmium. Lead determinations were made in the presence of pure copper nitrate with and without zinc and cadmium nitrates. No harmful effects were observed. In fact, the complete precipitation of copper free from lead was facilitated by the presence of amounts of these metals when not in higher percentage than the copper. Small amounts of iron do not interfere. It was impracticable to try varying quantities of all the metals. Zinc was most often applied in the proportions of brass, 25 to 35 percent. Larger quantities of iron and vanadium do not interfere with the complete deposition of copper, but they take the place of nitric acid, and unless the quantity of that acid be lowered there will not be the usual deposition of metal. Antimony and tin are so slightly soluble in nitric acid that they were not considered. Bismuth and arsenic interfere.

(b) Bismuth. If the quantity of bismuth be below 0.01 g. to 0.65 g. copper, the copper is more readily precipitated, contains nearly all of the bismuth, and has a much deeper red appearance than usual. The quantity of nitric acid may be increased to advantage. Instead of 4 cc., add 6 to 8 cc. More than 0.01 g. of bismuth renders the copper loose and granular, and above 0.1 g. it is black. The lead is not contaminated if the acid concentration is increased and the deposits are small, but above 0.01 g. bismuth, the lead deposits to some extent on the cathode.

(c) Arsenic. Varied amounts of arsenic in the form of arsenic acid were added to the electrolyte. Below 0.02 gram, an added amount of nitric acid prevented any contamination of the copper. The lead peroxide had a gray appearance, and deposited partly with the copper if the quantity of arsenic exceeded 0.02 gram.

CONCLUSION.

The aim of this work has been to facilitate the analysis of brass. Enough general data have been obtained to warrant the applica-

tion of the method to other alloys, provided they contain less than 2 percent of arsenic or bismuth. These metals in small quantities act as reducing agents, and require the addition of a little more nitric acid, whereas iron has an oxidizing effect, and allows of less nitric acid being used.

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DISCUSSION.

E. P. SCHOCH: In the introduction the author plainly states his intention to be the solution of the question "whether sulphuric acid would allow of copper being completely precipitated from a nitric acid solution while lead is being determined simultaneously on the anode." However, the experimental material presented in the paper does not directly attack this question at all and in no sense answers this question, because the author did not state that he ascertained experimentally that out of his mixtures *without sulphuric acid* the two metals cannot be deposited completely, while with the acid they can be co-deposited completely.

That the metals can be deposited out of such mixtures was shown several years ago by Sand (J. Chem. Soc., **91**, 397). The following is quoted from the page just referred to: "When it is desired to separate lead from a metal such as copper there is no need to acidify the solution strongly with nitric acid in order to prevent the deposition of metallic lead, if only the quantity of the metal is so great that all the lead is deposited on the anode a considerable time before exhaustion of the copper sets in. Thus an experiment was successfully carried out in which lead and copper were deposited in one operation from a solution containing sulphuric and nitric acids. . . . Measured quantities of the standardized copper sulphate and lead nitrate solutions were taken and 1 cc. of concentrated nitric acid added.

Cu taken 0.2474; Cu found 0.2476.

Pb taken 0.1383; Pb found 0.1386."

This method has been tested frequently in my laboratory, and the results obtained have been perfectly satisfactory.

It will be noticed that Mr. White used a smaller proportion of lead than was used by Sand; this, however, should facilitate the co-deposition of the metals, as was explained by Sand. The most important point of difference between Sand's method and that used by Mr. White is the slightly larger quantity of nitric acid. This greater amount of nitric acid could certainly have no harmful effect on the deposition of the lead, hence we are led to infer that this quantity of acid would prevent the deposition of the copper unless the sulphuric acid were present. Had Mr. White actually demonstrated by experiment that it was impossible to deposit the copper out of this mixture in the absence of sulphuric acid, then he would have proved the point which he has stated in his introduction to be his object. However, he did not do this, and, furthermore, it is well known that copper can be completely deposited out of electrolytes containing as much nitric acid as he used (see A. Fischer, Elektroanalytische Schnellmethoden, p. 106), and hence the sulphuric acid is not necessary.

It thus appears that there is absolutely nothing new in the paper before us.

A. J. White (*Communicated*): The title of my paper reads: "With the Rotating Anode." Mr. Sand's work was done with his special form of concentric gauze electrodes in a glass vessel; the term rotating anode as ordinarily used implies a stationary dish cathode. The distance between the electrodes makes a tremendous difference in the amount of acid necessary with copper and lead. Moreover, Mr. Sand does not claim simultaneous deposition of the two metals, as he clearly states later on the page quoted by Mr. Schoch, that first the lead is deposited, then the copper, by increasing the amperage from two to ten.

There is a great discrepancy in Mr. Schoch's argument dealing with the lead sulphate precipitate. To continue the quotation from Mr. Sand's article where Mr. Schoch omits, we have: "containing sulphuric and nitric acid. A precipitate of lead sulphate was present, which, however, dissolved as the lead was deposited on the anode. It should be mentioned here that in certain other

experiments attempts were made to separate metals from solutions containing precipitates which were not dissolved during the electrolytic operation. In nearly all such cases it was *not* found possible to wash the deposited metal free from the precipitate. Measured quantities of the standardized" etc., as Mr. Schoch quotes. Now, numerous experiments have shown that the redissolving of the lead sulphate possible with Mr. Sand's *gauze* electrodes is impossible with a platinum dish cathode. The precipitate driven against the solid surface adheres far too well to be washed off. I have clearly stated that the larger proportion of copper was necessary in order to prevent the precipitation of lead sulphate. Other substances can be introduced to hold up the lead when small amounts of copper are present. Considerable thorium nitrate takes the place of the copper and allows of a separation in any proportion.

Three different articles in the last two years dealing with the subject in hand show clearly that a standard method for analysis of copper alloys has not been reached. To quote from an article by E. Gilchrist and A. C. Cumming:[1]

"It is a matter of common experience that, in the electrolytic determination of copper in a solution containing nitric acid, it is impossible to deposit the last two or three milligrams under ordinary conditions. J. H. Stansbie has shown that this is due to the presence of nitrous acid."

Stansbie's valuable contribution to the knowledge of copper electrolysis was acknowledged in the paper under discussion. Hence it is unnecessary to prove that the two metals cannot be deposited completely without sulphuric acid, this being clearly shown in the opening paragraphs referring to Stansbie's work. Sand used the necessary amount of sulphuric acid in introducing his copper as copper sulphate, but he says nothing about the necessity of it.

A very recent article on the analysis of copper-tin alloys by Mr. Gemmell[2] gives developments of Mr. Sand's electrodes. The percentage of lead in the alloys tested was uniformly low, yet Mr. Gemmell claims that the lead sulphate was not completely dissolved, and requires separate weighing as $PbSO_4$.

[1] Chem. News, **107**, 217.
[2] Journal Society of Chemical Industry, **32**, 581 (1913).

Mr. Woiciechowski makes the statement that there is no practical exact method published for the determination of lead dioxide on a gauze cylinder, with rotating apparatus,[3] and proceeds to remove the anode containing the lead before completing the precipitation of the copper.

As the quotations from the above authors show the need for some experimental data bearing on the determination of copper and lead, the argument that "the experimental material . . . does not directly attack this question at all . . . because the author did not state that he ascertained experimentally that *without* sulphuric acid the two metals cannot be deposited completely, while with the acid they can be co-deposited completely," is very weak. First, because Stansbie was quoted to the effect that they could not without sulphuric acid, and, second, because all the experimental data given shows that they can.

[3] Metallurgical and Chemical Eng., **10**, 108 (1912).

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

RAPID REFINING OF COPPER WITH A ROTATING CATHODE

By C. W. Bennett and C. O. Brown.

Electrolytic refining of copper, as it is ordinarily done, is carried out with a current density of from 2 to 4 amperes per square decimeter of cathode surface. The amount of copper precipitated in one or two hours by a current of this density is small unless the cathode surface be very large.

For the purpose of demonstrating electrolytic refining as a laboratory experiment, the apparatus must be small, the amount of metal deposited must be appreciable, and the time must be limited. It has been shown previously[1] that if the cathode be rotated rapidly, the current density can be increased greatly, current densities up to about 425 amperes per square decimeter, having been used. With a small apparatus using current densities of this magnitude, sufficient amounts of copper for demonstration purposes, can be deposited in a short time, and appreciable amounts of the impurities may also be obtained as slimes.

It was thought advisable, therefore, to make a few tests on the rapid refining of copper to show that it could be done, and also with the idea of using it as a laboratory experiment. These experiments, of course, have very little practical value, and serve only to show that the same conditions obtain in rapid refining as in the commercial refining. They also give another instance of the rapidity of electrochemical work when the solution is stirred vigorously.

The conditions of these experiments, with the exception of the anodes, were practically the same as those obtaining in the previous work on this subject.[2] For this work, however, it was decided to design a more compact apparatus than that previously

[1] Trans. Am. Electrochem. Soc., **21**, 253, 268 (1912); Jour. Phys. Chem., **16**, 310 (1912).

[2] Trans. Am. Electrochem. Soc., **21**, 253 (1912); Jour. Phys. Chem., **16**, 294 (1912); **17**, 373 (1913).

used. The apparatus as actually used will be described in some detail.

The assembly drawing of the apparatus is shown in Fig. 1. The mechanical details, should any one desire to build the cathode, are shown in Figs. 2 and 3.[3]

Upon the base of a one-half horse power direct current motor

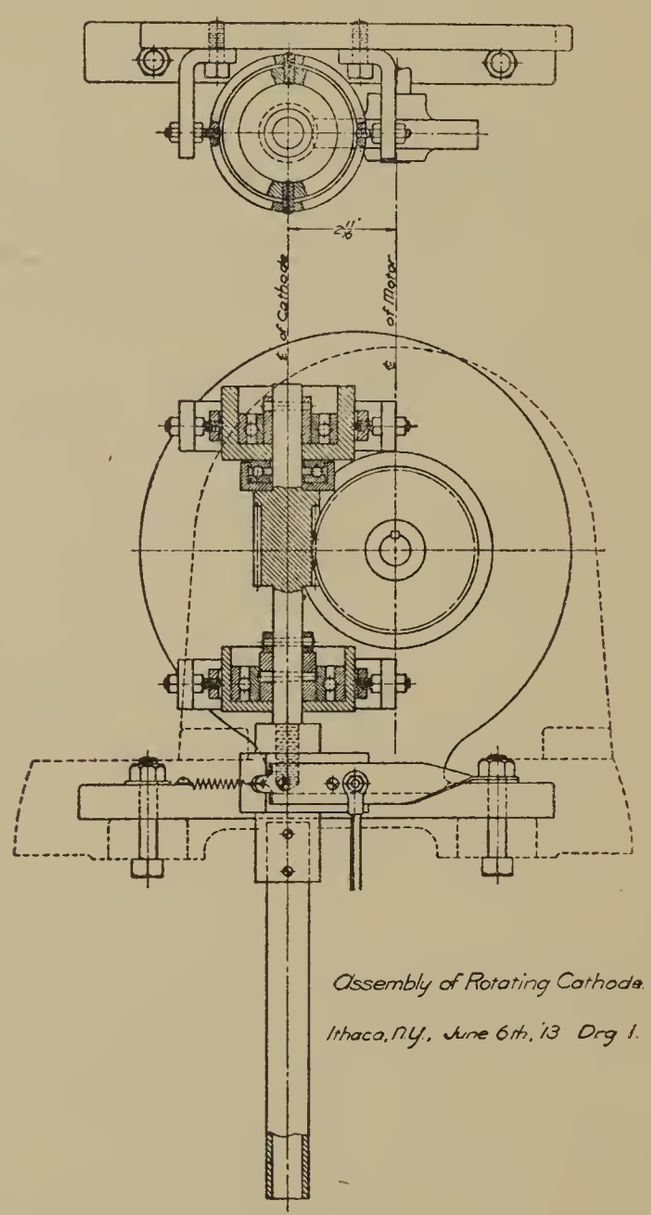

FIG. 1.

was mounted a face plate through which the shaft of the motor projected. Upon the shaft of the motor was mounted directly a wheel which has a worm gear cut on it. This worm wheel actuates a worm gear which was cut on the shaft, which was mounted vertically. This shaft operates in three bearings, two

[3] The cost of this apparatus should not be greater than eighty to ninety dollars.

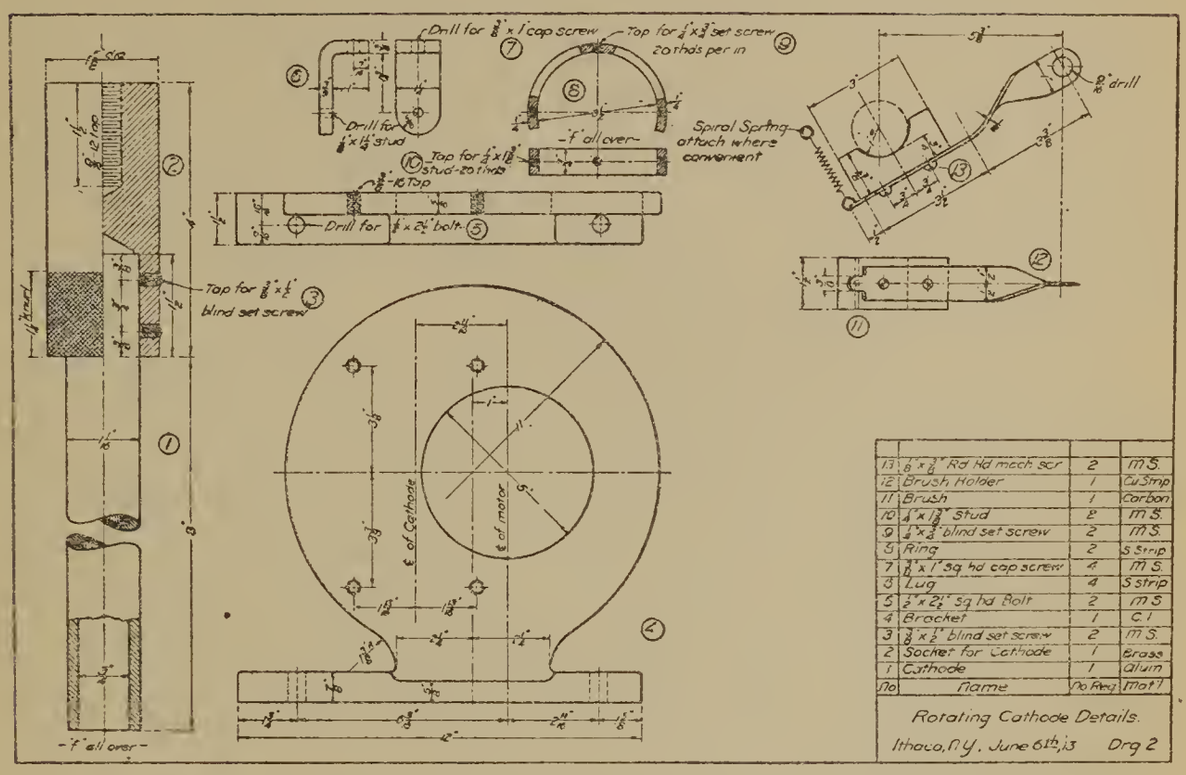
Drill for 3/8" x 1" cap screw
Tap for 1/4" x 3/8" set screw 20 thds per in
Drill for 1/4" x 1 1/2" stud
Tap for 1/4" x 1 1/2" stud - 20 thds
-f' all over-
Spiral Spring attach where convenient
3/8" drill
Drill for 1/2" x 2 1/2" bolt
Tap for 3/8" x 1/2" blind set screw
℄ of Cathode
℄ of motor
-f' all over-
13 3/8" x 3/8" Rd Hd mach scr 2 M.S.
12 Brush Holder 1 Cu Strip
11 Brush 1 Carbon
10 1/4" x 1 1/2" Stud 2 M.S.
9 1/4" x 3/8" blind set screw 2 M.S.
8 Ring 2 S. Strip
7 3/8" x 1" Sq hd cap screw 4 M.S.
6 Lug 4 S Strip
5 1/2" x 2 1/2" Sq hd Bolt 2 M.S.
4 Bracket 1 C.I.
3 3/8" x 1/2" blind set screw 2 M.S.
2 Socket for Cathode 1 Brass
1 Cathode 1 Alum
No Name No Req Mat'l
Rotating Cathode Details.
Ithaca, N.Y. June 6th, 13 Drg 2

FIG. 2.

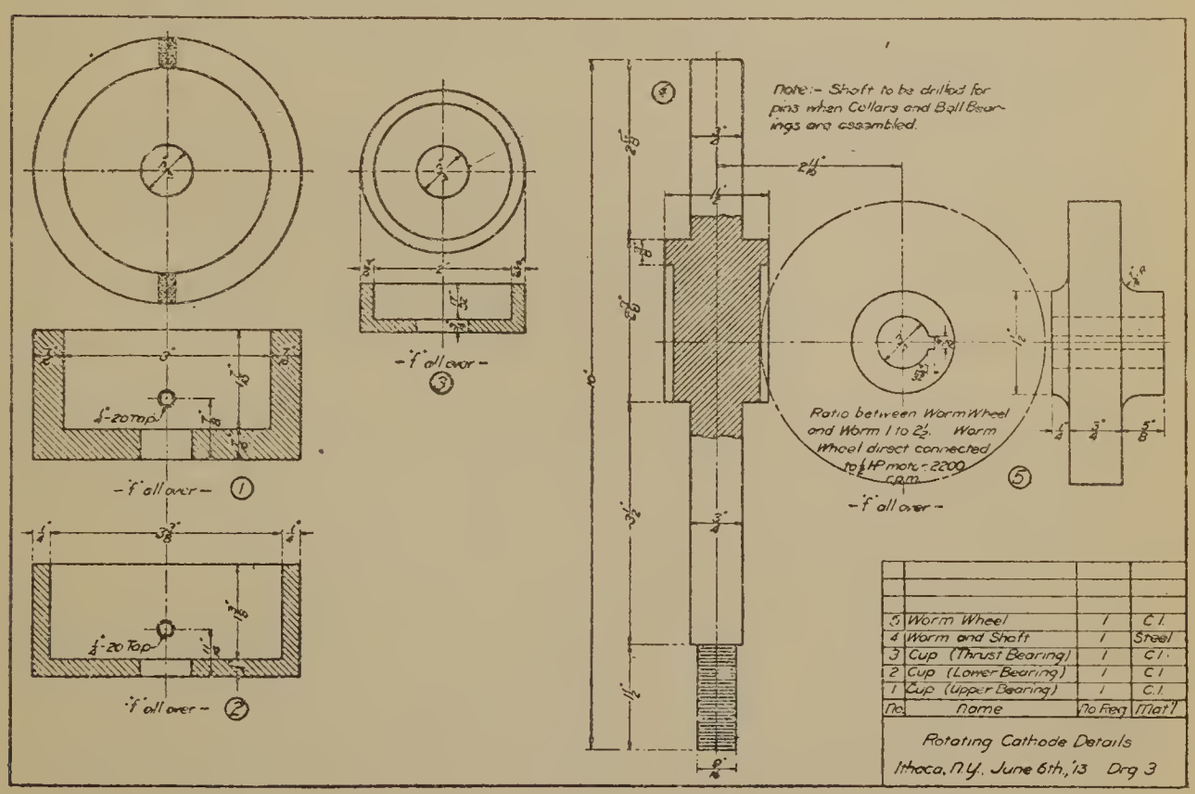
1/4-20 Tap
-f' all over-
-f' all over-
Note:- Shaft to be drilled for pins when Collars and Ball Bearings are assembled.
Ratio between Worm Wheel and Worm 1 to 2 1/2. Worm Wheel direct connected to 1/4 HP motor - 2200 r.p.m.
-f' all over-
5 Worm Wheel 1 C.I.
4 Worm and Shaft 1 Steel
3 Cup (Thrust Bearing) 1 C.I.
2 Cup (Lower Bearing) 1 C.I.
1 Cup (Upper Bearing) 1 C.I.
No Name No Req Mat'l
Rotating Cathode Details
Ithaca, N.Y. June 6th, 13 Drg 3

FIG. 3.

at the top and one at the bottom. The worm wheel turns clockwise, so that the shaft thrusts upwards. There is, therefore, a thrust bearing at the top, and also a radical bearing. The bearings are held at the top and bottom in iron cups, which in turn are held in steel rings, the steel rings being fastened to arms attached to the face plate. In this way, as can be seen by reference to the assembly drawing, adjustments of the bearing may be had in all directions. A flexible joint is obtained so that the bearing adjusts itself automatically.

As in the cathodes used previously, the shaft projects at the bottom where it is threaded, and screws into a brass connector. The bottom end of this connector is drilled to receive an aluminum pipe 1-1/16 inches outside diameter. This is held in the connector by set screws. The current is led away from the cathode at the connector by means of a carbon brush fastened with screws to a copper strip, which is connected to the base of the motor as shown. The pressure of this brush is adjusted by means of a spring.

The motor operates at speeds up to 2,200 R. P. M., so that the cathode with a 2½ : 1 gear may be run at practically any speed up to 5,500 R. P. M. The power required to turn the cathode has not been measured. It is, however, less than one-half horse power, and will depend, of course, upon the amount of metal on the cathode.

In these experiments the cathode extends into the solution about 12.5 centimeters, giving a cathode surface of about one square decimeter. If 70 amperes per square decimeter be used, which is a low current density for the rotating cathode, in one hour and a half, approximately 125 grams of copper will be deposited upon the cathode. This gives sufficient copper to show that the metal has been refined, and if the impurities in the anodes be present in high enough concentrations, sufficient slimes will be obtained to show tests for the several impurities.

In order to try out the method it was decided to use anodes containing silver as the only impurity. The first anode studied was, therefore, made up to contain approximately one percent. of silver. This amount of silver was added so that within the time set for the experiment, sufficient metal could be obtained to make a silver bead, thus doing away with the danger of dis-

appointing the student by his not being able to obtain appreciable amounts of the impurity. If the process works efficiently, approximately 1.2 grams of silver should be obtained as slimes, in one and one-half hour's run.

In the other anodes studied the metals were added in the proportion to make one percent. There is practically no loss in casting with any of the metals, save zinc. About 5 percent extra metal should be added in this case. Two anodes of each composition were cast, smooth, solid castings being required. If the metal is porous, the slimes are hard to remove.

The first test was on copper containing one percent silver. The anodes were connected in multiple, one on each side of the cathode. The current used was 65 amperes and the run was short. The metal deposited was tested and, as was anticipated, was found to contain silver. On account of the rapid rotation and vigorous stirring, the slimes, of course, do not stick to the anode, but are diffused mechanically throughout the solution and are, therefore, occluded in the deposit mechanically.

Diaphragms consisting of heavy unbleached cotton cloth were then prepared. These were made by folding the cloth and sewing it so as to form bags which fit closely to the anodes themselves. The solution used, as in the previous work, was 12 percent copper sulphate with 15 percent sulphuric acid. The cloth was found to stand up well for several days in this solution.

The next run, with the anodes enclosed in the cloth bags, was made under the same conditions. The current was 65 amperes, and the run lasted a little more than an hour, 90 grams of copper being deposited. The voltage drop across the cell was 5 volts. The deposited copper in this case was pure and gave no tests for silver.

The slimes were recovered by burning the diaphragms and treating the ashes with nitric acid, filtering and depositing the silver as chloride and reducing, finally obtaining the silver as a bead before the blowpipe, or by depositing electrolytically from the nitrate solution. A good bead of silver was obtained, wholly sufficient for demonstration purposes.

It has been stated by Kiliani[4] that the silver present as impurity in copper anodes, goes into solution if a neutral copper sulphate

[4] Kiliani, Borchers, Electric Smelting and Refining.

solution is used as electrolyte. The reason for this is not at all evident. It was thought that under the conditions of this experiment a large amount of silver could be obtained in the electrolyte in a short time, since larger current per unit volume of solution was passed.

A run was therefore made using an anode containing silver in a copper sulphate solution without the addition of acid. A run was also made on a copper sulphate solution to which ammonium hydroxide was added until it was neutral to litmus paper. After running for a considerable length of time in both cases, the solutions and the deposited copper were tested for silver. No silver could be found in either, so that there is some doubt as to the validity of the statement referred to above.

The second anode studied consisted of approximately the following composition:

Copper	97 percent
Silver	1 percent
Arsenic	1 percent
Bismuth	1 percent

The current and voltage were the same as in the preceding experiment, the electrolysis being run for about one hour and three-quarters. In all of these experiments the speed of rotation was between 5,500 and 6,000 R. P. M.; 125 grams of copper was obtained in this case. The deposit was excellent in appearance, and showed only a trace of arsenic by the Marsh test. No silver and no bismuth could be detected in the metal. Slimes were examined, as before, by burning the diaphragms and depositing the metals. Tests for bismuth and arsenic were given, and the silver bead was obtained as before.

The last anode studied contained representatives of all the impurities found in commercial anodes, although present in larger quantities. Since this anode was intended to contain all the metals for demonstration purposes, a quantitative analysis of it was made. The analysis is difficult and not essential.

Unless quantitative results are desired it is sufficient to add all of the metals in the proportions desired with the exception of zinc. Five percent of this metal should be added in excess. For those who wish to make the analyses the following refer-

ences may be of interest.[5] The anode was found to have the following composition:

Metal	Percent.
Copper	91.00
Silver	1.09
Lead	1.16
Iron	1.10
Nickel	1.14
Bismuth	1.15
Arsenic	1.20
Zinc	1.17
Carbon	0.70
Total	99.71

The runs on this anode were quite as successful as the previous ones, the conditions of the runs being the same—current, 65 amperes; voltage, 5 volts; time, about 1.75 hours; copper deposited, about 130 grams. The deposit was found to be quite up to the standard of average electrolytic copper.

Of the impurities in this anode, silver, lead, bismuth and arsenic were found in the slimes. Qualitative separations were made of the metals found here, and the usual silver bead obtained at the end of the run. Tests for iron, nickel and zinc were obtained from the solution, although it takes more care to detect these metals in the solution without concentration, and the test is not so satisfactory as in the case of the slimes.

No quantitative tests were made on the deposited copper, but qualitative tests indicate that it would require very accurate analyses to detect any variation from 100 percent copper. Quantitative tests were not made on the slimes to determine how efficient the recovery was. Some of the slimes, of course, stick to the anodes and are therefore hard to obtain, so that the quantitative tests would not have meant anything had they been run.

In conclusion, electrolytic copper refining can be demonstrated in the course of an hour if a rotating cathode be used. The anodes may contain impurities usually found in commercial anodes, the demonstration being more striking if the impurities are present in amounts approximating 1 percent each. The anodes should be contained in a diaphragm, and the slimes may

[5] Jour Am. Chem Soc., **16**, 185 (1894); **17**, 714 (1895); **19**, 24 (1897); **22**, 292 (1900); **24**, 699, 1082 (1902); **27**, 313 (1905); **29**, 607 (1907); Zeit. anal. Chem., **46**, 29 (1907).

be removed partially with the diaphragm, and the remainder by means of a stiff brush and water from a wash bottle. The deposited copper may be stripped off and sampled if aluminum cathodes are used.

Arsenic shows the largest tendency to contaminate the refined copper. It requires, however, the delicate Marsh test to detect this metal.

The statement that silver dissolves in neutral copper sulphate solution is probably an error.

Electrochemical Laboratory,
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A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

SOLID, THICK DEPOSITS OF LEAD FROM LEAD ACETATE SOLUTIONS.

By Frank C. Mathers.

Any soluble lead salt will give a solid, dense, coherent electrolytic deposit if the proper addition agents are used. Those solutions which do not give good deposits will do so when the proper addition agents are introduced. This theory cannot as yet be proved in all cases, but this paper gives the experimental proof in the case of lead acetate solutions. The idea has been advanced very strongly by Betts[1] that "the basis of the lead depositing electrolyte should be a strong—*i. e.,* strongly dissociating acid, and practically non-oxidizing, at least so far as the cathode is concerned. *No good deposit has ever been obtained when the basis of the electrolyte is a weakly-dissociating acid.*"

REVIEW.

Glaser[2] claimed to have obtained solid deposits from a solution of lead and potassium acetate, but no one has been able to duplicate his results. Snowden[2] electrolyzed lead acetate and acetic acid solutions with a stationary and also with a rotating cathode. He obtained a crystalline, adherent deposit. He found that the addition of glue was very beneficial. These deposits were very thin. Senn[2] said that the addition of glue to lead acetate baths failed to produce dense deposits. He concluded that the fluosilicate radicle had some specific property which assisted in the formation of solid deposits. Bancroft[3] says that "if a lead acetate solution is made as strongly acid as the lead fluosilicate solution, glue or gelatine does cause a good deposit." He performed an experiment upon a solution containing 10 percent lead

[1] U. S. Pat. 679,824 (1901); Trans. Am. Electrochem. Soc., **17**, 272 (1910).
[2] Trans. Am. Electrochem. Soc., **23**, 156, 163, 172 (1913).
[3] Trans. Am. Electrochem. Soc., **21**, 333 (1912).

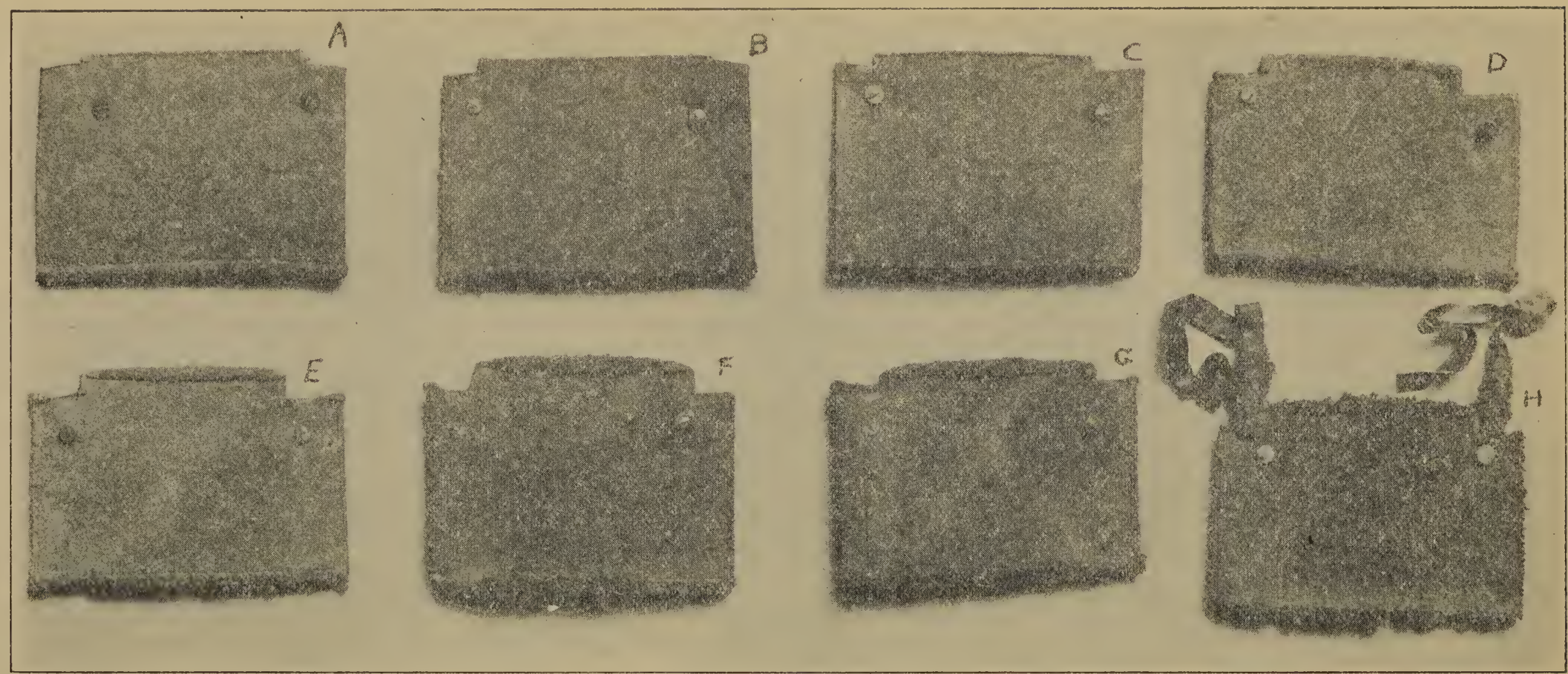


FIG. 1.

These baths contained 10 percent lead acetate, and varying amounts of other substances as follows:

A. 16 percent ammonium perchlorate, 5 percent acetic acid and 1 percent peptone.

B. 8 percent ammonium perchlorate, 5 percent acetic acid and 1 percent peptone.

C. 4 percent ammonium perchlorate, 5 percent acetic acid and 0.5 percent peptone.

D. 3 percent ammonium perchlorate, no acetic acid, and 0.25 percent peptone.

E. 2 percent ammonium perchlorate, no acetic acid, and 0.25 percent peptone.

F. 1 percent ammonium perchlorate, no acetic acid, and 0.25 percent peptone.

G. ½ percent potassium perchlorate, no acetic acid and 0.25 percent peptone.

H. 4 percent ammonium perchlorate, 5 percent acetic acid and 1 percent glue.

acetate and 5 percent acetic acid, and found a marked beneficial effect, due to the addition of 1 percent of glue. This diversity of opinion concerning the effect of the glue in lead acetate solutions may be accounted for by two different definitions of a "good" deposit. One experimenter secured from this solution a thin deposit which was smooth and "good," while another experimenter tried to make a thick deposit which was rough, crystalline and not "good."

MANIPULATION.

In the experiments described in this paper, beakers containing 200 c.c. of the electrolyte were used as electrolyzing vessels. A cathode, 4.2 by 6 cm., made of thinly-rolled lead was suspended from a glass rod by two narrow strips of lead. Two anodes, 3.5 by 4 cm., were suspended in each beaker. At the time of casting the anodes a piece of wood was held in the center of each mold, so that there would be a hole in each finished lead anode. Small copper wires passing through these holes were used to hold the anodes in place and to make electrical connection. The copper wires never dissolved appreciably in the acetate bath as long as any lead remained. The anodes were each wrapped in a filter paper to prevent anode slimes from reaching the cathodes. Storage batteries furnished the current, hence electrolysis was continuous. No arrangement for stirring or circulating the solutions was made, which accounts for the thickened deposits upon the lower parts of the electrodes.

EXPERIMENTS WITH DIFFERENT ADDITION AGENTS.

Ammonium Perchlorate and Glue:—Glue is a cheap addition agent which gives excellent deposits from some strongly acid solutions. It is especially good in fluosilicic acid solutions, but it is of little value in perchloric acid solutions. Glue greatly improves the deposits from lead acetate solutions containing free acetic acid. This fact has already been noted by Snowden and Bancroft as mentioned in the introduction. These deposits are smooth on the flat parts of the cathodes, but loose crystals form upon the edges, so that thick "good" deposits cannot be made in this way. By the addition of 4 percent of ammonium perchlorate to one of these baths the formation of long, loose crystals upon

the edges of the cathode almost ceased, being replaced by rough, fairly solid projections. The deposit was very finely crystalline. As electrolysis continued, pointed crystals or projections grew from the center of the plate, and finally the deposit became entirely unsatisfactory. This cathode is shown in Fig. 1. This bath could not be used for plating or refining purposes. Samples or pictures or these cathodes do not show all their faults, because many of the loose crystals fall off during handling.

Since pure gelatine gives such a poor deposit as compared to glue, the beneficial action must be due to some other substance than "gelatine." However all attempts to change gelatine into a satisfactory addition substance failed.

Ten gm. gelatine, 4 gm. sodium hydroxide, and 100 c.c. of water were digested with 0.5 gm. pancreatin at 35°. Portions of this solution containing one gram of gelatine were removed and added to baths at intervals beginning after 36 hours and extending to 407 hours. The deposits were less satisfactory than with untreated gelatine.

Hydrolysis of gelatine by boiling with aqueous acetic, hydrochloric, or nitric acid gave a material which produced black spongy non-adherent lead.

There seems to be no way of changing glue to improve its properties as an addition agent for use in lead acetate baths.

Ammonium Perchlorate and Peptone:—Peptone which was found to be very beneficial in lead perchlorate baths[4] proved to be the best addition substance for use in lead acetate solutions containing ammonium perchlorate. In solutions containing no perchlorate the deposits were crystalline and non-adherent, like those obtained by the use of glue.

Effect of the Quantity of Ammonium Perchlorate:—Fig. 1 shows cathodes from solutions containing varying amounts of ammonium perchlorate. A cathode obtained with glue instead of peptone is shown for comparison. The cathode with 2 percent potassium perchlorate is almost as good as the corresponding one with ammonium perchlorate. No acetic acid was used in the solution containing 1, 2, and 3 percent ammonium perchlorate. Free acetic acid was used in all the earlier experi-

[4] Mathers, Trans. Am. Electrochem. Soc., **17**, 261 (1910).

ments, in deference to the ideas of other experimenters that good deposits could be obtained only from strongly-acid solutions. The very rough deposit with 1 percent ammonium perchlorate shows the very great importance of this salt in the solution. Four percent of ammonium perchlorate is a suitable amount to use. The cathode deposits were brittle.

Effect of Acetic Acid:—A comparison of the cathodes in Fig. 1 shows that slightly better deposits were obtained from solutions which did not contain any free acetic acid. The addition

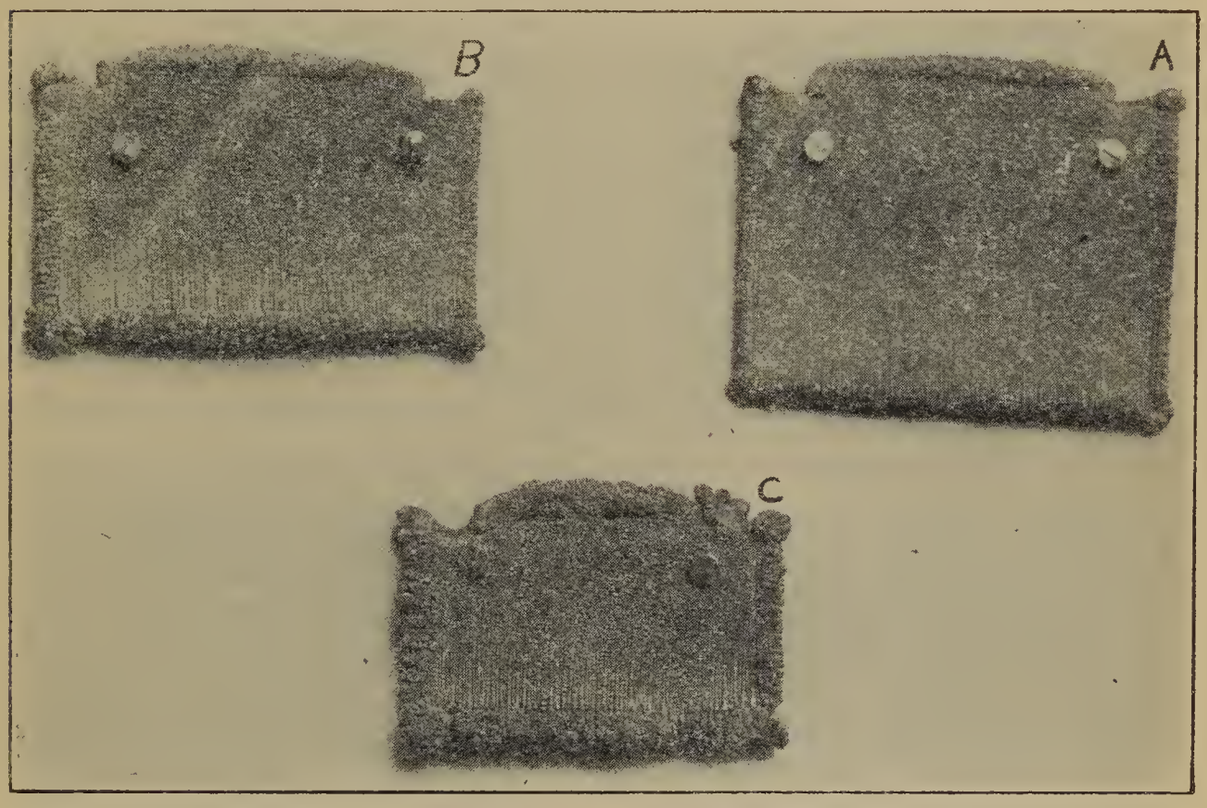


FIG. 2. EFFECT OF CURRENT DENSITY.

These solutions contained 10 percent lead acetate, 4 percent ammonium perchlorate, no acetic acid and 0.25 percent peptone. The current densities at the cathode were:

A. 0.5 amp. per sq. dm. (4.5 amp. per sq. ft.)
B. 0.63 amp. per sq. dm. (5.7 amp. per sq. ft.)
C. 1.0 amp. per sq. dm. (9 amp. per sq. ft.)

of 1 percent of free acid seemed to be without effect. Even the use of 5 to 10 percent of free acetic acid showed only slightly injurious action upon the deposit. Free acid is objectionable in that it dissolves the lead straps which hold the cathode in place. All of the baths prepared from lead acetate, $Pb(CH_3.COO)_2.3H_2O$, were acid in reaction. However, a solution of tri-basic lead acetate which was alkaline to litmus gave almost as good

a deposit as the slightly-acid solutions. All of this shows the erroneousness of the older idea that only strongly-acid lead solutions give coherent deposits.

Effect of the Quantity of Peptone:—Witte's peptone is not completely soluble in lead acetate solutions, so there was always a slight precipitate in the baths, which might have been the cause of some of the unevenness of the deposits. There was a tendency for large amounts of peptone to make the deposits more brittle and a little less smooth. Cathodes with 1 percent and with 0.25 percent peptone are shown in Fig. 1. It seemed that 0.25 percent peptone was the best quantity to add. In one experiment, 118 gm. of lead was deposited in excellent condition with the addition of 0.5 gm. peptone. This would be at the rate of 8 pounds of peptone per ton of lead deposited. It was necessary to use more peptone than in the case of the lead perchlorate baths. In these acetate solutions 0.05 percent of peptone did not prevent the formation of a fringe of crystals on the edges of the cathodes.

Effect of Current Density:—Fig. 2 shows cathodes obtained with varying current densities. While most of the experiments described in this paper were run at a current density of 0.4 amp. per sq. dm. (3.6 amp per sq. ft.) a current density as high as 0.7 to 0.8 amp. might have been used. As the current density increases the cathodes become rougher, but not more crystalline.

Effect of Concentration of Lead Acetate:—Fig. 3 shows cathodes obtained from solutions containing varying amounts of lead acetate. With circulation of the solution a good deposit might have been obtained from the bath containing only 2 percent of lead acetate. This very dilute solution gave a bad deposit only upon the upper part of the cathode, where the lack of stirring first shows its effect. The deposits from the solutions containing 6 and 8 per cent lead acetate seem as good as the one from the 10 percent lead acetate bath.

OTHER SALTS IN PLACE OF AMMONIUM PERCHLORATE.

Sodium chlorate from 4 to 24 percent, *ammonium acetate* from 4 to 28 percent, *manganese acetate* 4 per cent, *zinc acetate* 4 percent, and *barium acetate* 4 percent gave poor deposits. Sodium

chlorate gave deposits which were better than with the others, but which were of no value.

Sodium perchlorate, 4 percent, gave as good deposits as the ammonium perchlorate. The latter salt happened to be at hand in large amounts, so it was used in most of the experiments. One sample of sodium perchlorate which contained sodium chloride as an impurity did not give good deposits. This trouble was ascribed to the chloride, which always seems to have an injurious effect.

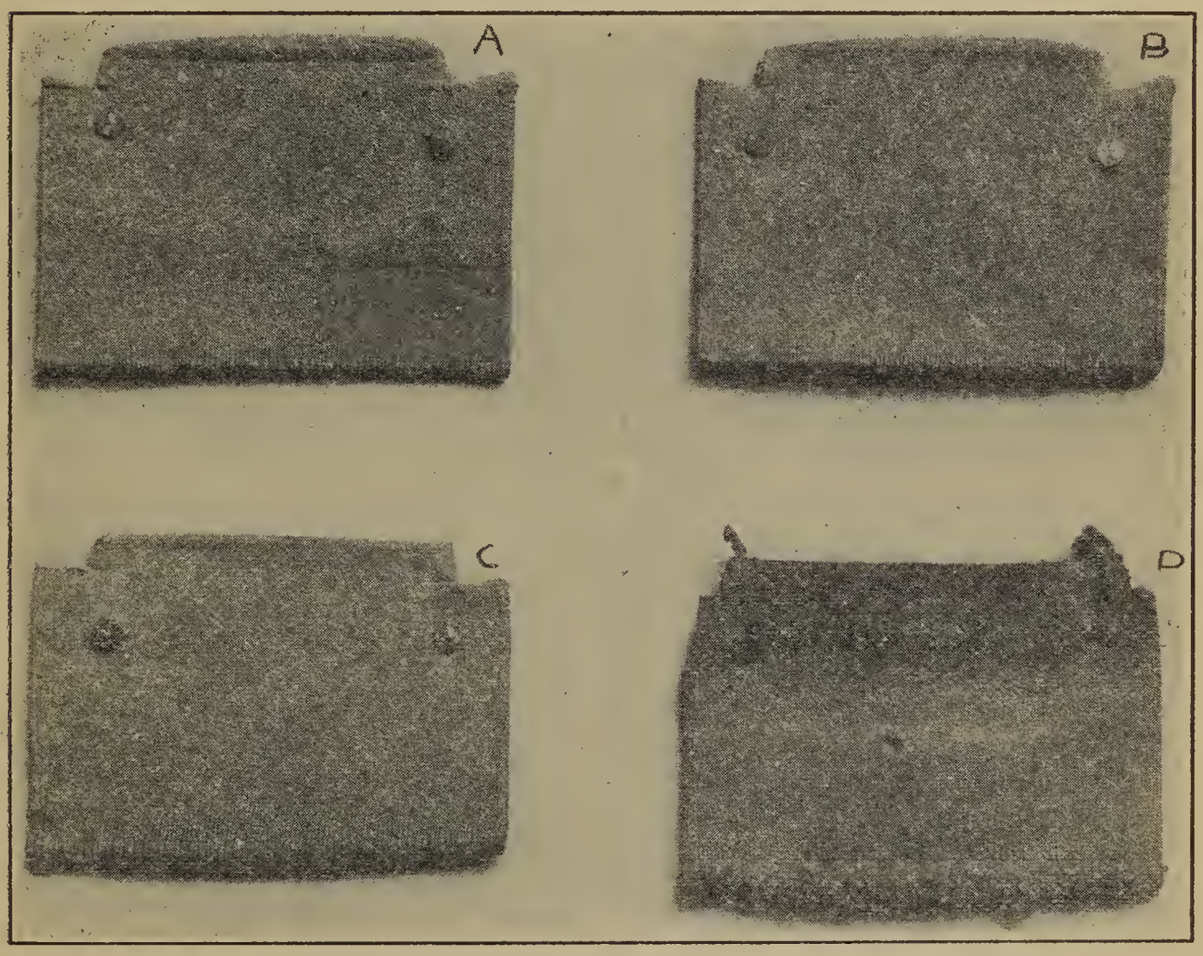


FIG. 3. EFFECT OF CONCENTRATION OF LEAD ACETATE.

These solutions contained 4 percent ammonium perchlorate, no acetic acid, 0.25 percent peptone and varying amounts of lead acetate as follows:

A. 8 percent lead acetate.
B. 6 percent lead acetate.
C. 6 percent lead acetate.
D. 2 percent lead acetate.

Sodium naphthelene sulphonate, 2 percent, when added to the bath formed a heavy precipitate. Five percent of acetic acid did not redissolve the precipitate. This bath, after filtration, gave excellent deposits of lead.

Sodium fluosilicate and sodium ethyl sulphate did not prove satisfactory, but this may have been due to faulty experimental conditions.

Lead chloride was not satisfactory. Of course the concentration was low, but chlorine ions always seemed to injure the deposit.

Ferric acetate, chromium acetate and *aluminum acetate,* with or without peptone or gelatine and acetic acid, did not give good deposits.

Potassium perchlorate is not suitable because it is so insoluble that a 2 percent solution, when a little evaporation takes place, will deposit crystals which produce rough places on the cathode. A 1 percent solution is too dilute to be very effective.

OTHER ADDITION SUBSTANCES.

Extract from Feathers:—Promising results, but nothing entirely satisfactory, were obtained by the use of extracts from feathers. Keratin, a material used in pharmacy work for coating enteric pills, is prepared by heating feathers in glacial acetic acid. This keratin is soluble in glacial acetic acid, but is insoluble in water and dilute acids. There are other substances present which remain in solution even in lead acetate solution, and which have a beneficial effect upon the lead deposits.

Many methods of treating the feathers were tried. One of the most successful was the following: One and a half grams of feather quills was heated in 10 c.c. of glacial acid until disintegration of the feathers had taken place. Most of the acetic acid was then removed by heating on a water bath. The water extract from this waxy residue had very little effect upon the lead deposition. The residue, which was insoluble in the water, was dissolved in 5 c.c. glacial acetic acid, and the solution thus obtained was added to the lead bath. Of course, the bulky precipitate which formed was removed by filtration. The deposits upon the cathode were better than with glue, but thick smooth deposits could not be obtained.

Boiling the feathers with the acetic acid for 188 hours and treatment as above gave no better results.

Feathers readily dissolve in sodium hydroxide, but the solu-

tions thus obtained, after neutralization with acetic acid, caused the lead deposits to be very black and spongy.

In another experiment, feathers were boiled in 1 : 1 hydrochloric acid until solution was complete. Only a few minutes heating was required. The hydrochloric acid was removed by heating on a water bath. Addition material prepared in this way produced great volumes of black, spongy, non-adherent lead, which fell from the cathode. Often the beaker would be a fifth full of this mud. After many hours the effect of the addition substance disappeared, and the deposit showed small bright crystals.

Solution of the feathers in dilute sulphuric acid did not give any satisfactory results.

Many different concentrations of acids and alkalies and periods of heating were tried in the preparation of the extract from feathers, but the results were never satisfactory. I do not believe that any satisfactory addition substance can be prepared from feathers.

Casein:—In the search for a material similar to glue, casein was selected for trial because it is a cheap, nitrogeneous animal substance. The great problem was to change the casein, which is insoluble in aqueous acid solutions, in some way, so that enough of it will remain dissolved in the lead acetate solution to give a good deposit. The addition of the casein solution, no matter how it was prepared, always produced a heavy precipitate in the bath, which was removed by filtration before electrolysis. A summary of the deposits obtained with casein shows that extraction of the casein in water, aqueous sodium acetate, aqueous acetic acid, alkali and then aqueous acetic acid, glacial acetic acid, or anhydrous glycerine, gave material which affected the deposits in about the same way. None of the addition substances prepared in these ways was satisfactory, although all showed marked beneficial action. Extraction of the casein with hydrochloric or sulphuric acid gave material which caused the lead to deposit in the form of black, loose sponge. Some cathodes are shown in Fig. 4.

Five grams of casein, 10 c.c. hydrochloric acid and 100 c.c. water were boiled for 18 hours. The hydrochloric acid was then

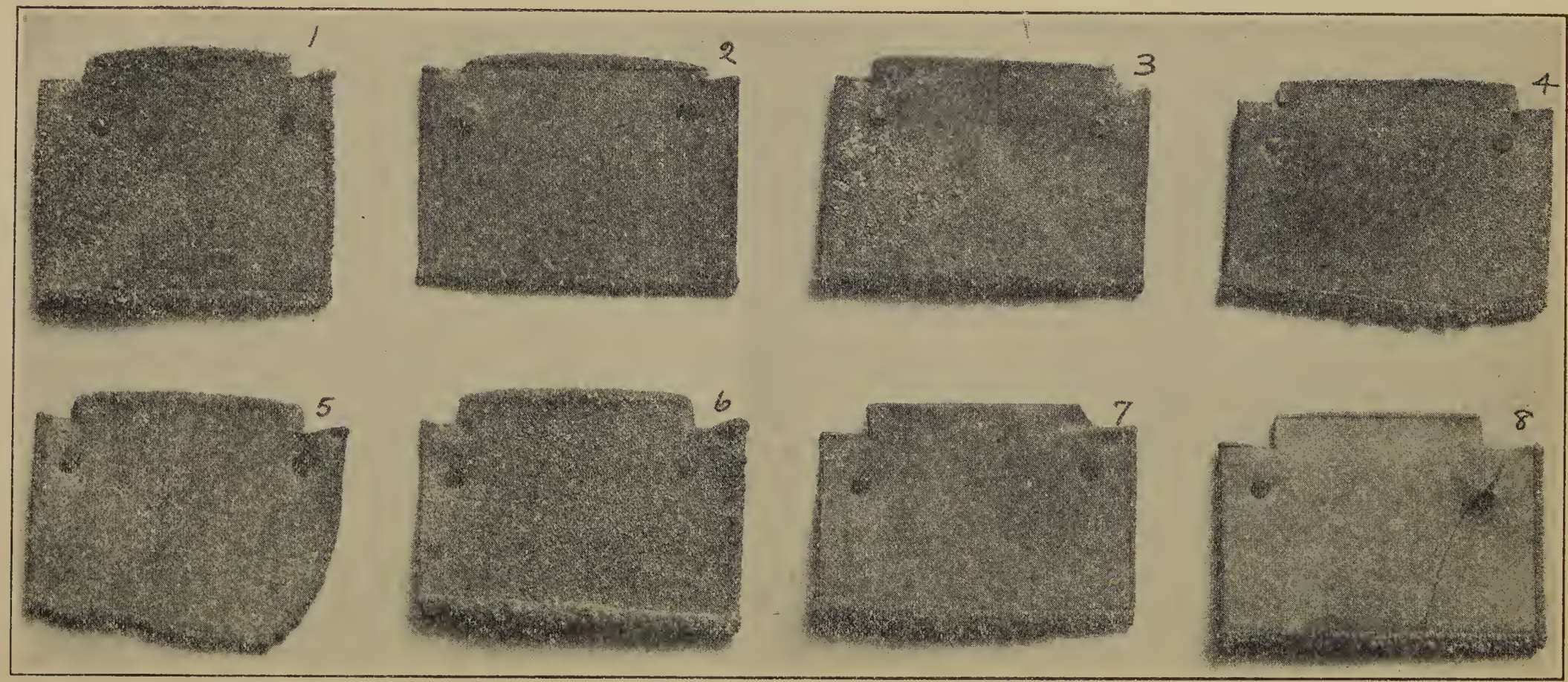


Fig. 4. Casein as Addition Substance.

removed by evaporation upon a water bath. The cathode deposit from a bath containing enough of this solution to represent 0.35 percent of casein was very loose and black. A few dark feathery crystals were formed. As the electrolysis continued the deposit became more metallic in color. The bottom of the beaker was covered with a deep layer of the black spongy lead which dropped from the cathode. After 15 days there was no indication of the deposit becoming solid.

Casein dissolved in boiling sodium hydroxide is not completely precipitated by acetic acid. Five grams casein, 5 grams sodium hydroxide and 50 c.c. water were boiled for 2 hours. The alkali was neutralized with acetic acid. Cathode 1 was from a bath containing enough of this solution to correspond to 0.35 percent casein. The deposit was bright and solid, but rough.

Five grams of casein and 60 c.c. of glacial acetic acid were heated in a water bath for 72 hours. This mixture could not be heated in a sand bath on account of the scorching of the casein which stuck to the sides of the flask. Cathode 2 was from a bath containing 0.5 percent of this casein and 6 percent of acetic acid.

Cathode 3 was made with 0.5 percent casein, prepared by boiling casein with 5 c.c. acetic acid and 100 c.c. water for 20 hours. The use of stronger acetic acid or the presence of lead acetate during the boiling of the casein made no difference upon the results.

Cathode 4 was from a bath containing 0.5 per cent. casein, prepared by boiling 1 gm. of casein in 10 c.c. of acetic acid for 10 minutes. This was added to the bath, which was heated to boiling before being filtered. The deposit was rougher than the others.

Cathode 5 was with 5 percent acetic acid and 0.15 percent casein, prepared by boiling 3 gm. casein with 100 c.c. glacial acetic acid for 15.5 hours on a sand bath.

Cathode 6 was with 0.5 percent casein, prepared by boiling in acetic acid for 70 hours.

Cathode 7 was with 2.5 percent casein, which had been prepared by heating 5 gm. casein in 5 c.c. acetic acid and 100 c.c. water for 49 hours in a water bath. This is probably the best cathode obtained by the use of casein.

Cathode 8 was from a solution containing 2.5 percent casein, prepared by boiling in 100 c.c. water for 49 hours. Enough casein had dissolved to impart a salmon color to the solution. No acetic acid was present at any tme.

In another experiment, 5 gm. casein was boiled in 40 gm. sodium acetate and 100 c.c. water for 100 hours. The deposit was like the others.

A bag filled with freshly precipitated casein was suspended in one bath. It did not prevent the formation of crystals. Apparently not enough casein went into solution to have much effect.

Five gm. casein was heated in glycerine until a thick solution had been made. This was poured into water, and the solution thus formed was added to the bath. The deposit was about as before.

Ten gm. of dry casein was heated in an air bath at 140° for several hours. The water extract from this casein was added to a bath. Again the cathode was like the others.

Casein Modified by Digestion With Enzymes:—Three portions of 10 gm. dry casein in 2000 c.c. water were digested at 35° with 0.5 gm. pancreatin, and 8 gm. sodium hydroxide, with 0.5 gm. pepsin and 45 c.c. hydrochloric acid, and with 0.5 gm. pepsin and 86 c.c. acetic acid respectively. Portions of these solutions corresponding to 0.25 percent casein were removed and used in lead baths. Enough acetic acid was added to neutralize the sodium hydroxide in the pancreatin digestion before the beginning of the electrolysis. No effort was made to remove the hydrochloric acid in the case of the pepsin digestion. Solutions were removed for experiments when the digestion had run 48 hours, and at intervals thereafter until the 487th hour. Both the pepsin digestions produced deposits which were dark, spongy and often non-adherent. The hydrochloric acid digestion was much worse than the acetic acid digestion. The pancreatin digestion gave deposits which were very solid, but very rough. These deposits were not equal to those obtained by merely heating the casein in acetic acid.

Tannin and Similar Substances:—Tannin or tannic acid has been found to have a valuable effect in certain baths. The difficulty in this work was that the lead acetate in the solutions precipitated the tannin. The heavy precipitate of lead tannate was

removed by filtration in each case before electrolysis. One percent of tannin and 5 percent of acetic acid produced a deposit upon the cathode which was black, spongy and non-adherent. 0.1 percent of tannin gave about the same results. 0.05 percent of tannin gave feathery crystals which looked as if no addition substance had been added. Tannin prepared by boiling for 186 hours with glacial acetic acid still gave the same black spongy lead.

One percent of pyrogallol and 5 percent of acetic acid gave light feathery crystals, although there were some smooth places upon the cathodes.

Meat Extracts:—One percent of meat extracts gave a black spongy non-adherent deposit. The same black deposit was formed during the entire 15 days during which the electrolysis continued.

Three grams of meat extract and 300 c.c. acetic acid were boiled for 15.5 hours. The cathode deposit was adherent, but very rough.

Two grams of meat meal or scraps was boiled with 100 c.c. water for several hours. The deposit was good at first, but gradually became worse, and finally became black and non-adherent.

Vegetable Extracts:—A heavy voluminous precipitate is always formed when these extracts are added to the lead acetate solutions. This is an objection to the use of extracts.

One percent of licorice extract produced a finely crystalline deposit; 0.2 percent gave a dark loose spongy mass.

One percent of lobelia extract gave a finely crystalline deposit.

One percent. soluble starch, prepared by heating with glycerine, gave a soft, black mushy deposit.

One percent of flax-seed was soaked in warm water over night, and the thick mucilage mixture was added to a lead bath. This is the addition substance that was patented by Siemens and Halske. The deposit, in the beginning, was bright and crystalline, but it gradually changed until it became black and non-adherent. The extract from ground flax-seed acted in the same way.

Ground slippery elm bark, which forms a mucilage with water gave results similar to the flax-seed.

Essential and Fatty Oils:—Clove oil improved the deposit a little. 0.5 percent clove oil in the form of an emulsion, prepared by using powdered acacia, gave a bright, dense but crystalline deposit. A smooth deposit of much thickness could not be obtained. The deposit seemed to be improved by the presence of free acetic acid.

Turpentine, when added in the form of an oil or as an emulsion, was without good effect.

Eucalyptol and safrol were of no value.

Emulsions of cod liver oil, oleic acid and paraffine oil prepared with powdered acacia, and almond oil in the case of the paraffine oil, were used to the extent of 2 percent without marked beneficial action. Again the presence of acetic acid seemed to improve the deposits a little.

Carbon bisulphide and benzol gave exceptionally dark, loose spongy deposits.

Gums:—Gum guaiac and gum gambir, 1 percent, gave deposits which were bright at first, but soon became black and non-adherent.

OTHER ELECTROLYTES.

Lead nitrate, 40 percent, and sodium nitrate, 10 percent, without addition substances gave a fairly smooth deposit on the flat part of the cathodes, but projecting crystals formed on the edges of the plate. The copper wires which made connection to the anodes were dissolved.

Lead acetate, 55 percent, and potassium acetate, 20 percent, without addition substance, gave a mass of light fluffy glistening leaflets or plates over the entire cathode. Glaser[5] claimed that these solutions would give solid, smooth deposits.

Lead chloride, 5 percent, and sodium chloride, 30 percent, did not give a solid deposit with casein as an addition substance.

Lead sulphate 10 gm., ammonium acetate 50 gm., water 100 c.c., never formed a clear solution. Electrolysis of this solution was not attempted, since it seemed that too small an amount of lead had dissolved.

[5] Z. Electrochem., 7, 365 (1900).

All of these halogen and nitrate solutions very readily attack the copper in contact with the lead anode, so that refining of lead from such solutions would be impossible.

Indiana University,
Bloomington, Indiana.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

EFFECT OF LIGHT ON DECOMPOSITION VOLTAGE

By Alan Leighton.

The following experiments were undertaken to determine the effect of light on the decomposition voltage of copper sulphate solutions. A mercury vapor lamp, enclosed in a tube, was placed in the copper sulphate solution between the electrodes. By means of a rubber cloth it was possible to shield either electrode from the light, or both electrodes if necessary. An ammeter was placed in series with the electrolytic cell, and a voltmeter was connected to the terminals of the cell. This arrangement[1] does not give the true decomposition voltage because the voltage drop through the solution is included. Since the electrodes were always kept in the same position, the error is practically constant throughout each series, and is negligible, because we were interested in the change of voltage produced by turning on the light. From the experiments of others it seems justifiable to assume that light causes no marked change in the conductance of a copper sulphate solution except in so far as the heat from the lamp may change the temperature of the solution.

Owing to the relatively high resistance of the solution it was not found practicable to determine the decomposition voltages by plotting voltages against current. The error due to the personal equation is much too large. It was found better to keep the voltage constant for a while, and then to determine by inspection whether copper had or had not been precipitated at the cathode.

[1] The current flowing through the voltmeter also flows through the ammeter.

In Table I are given data for the copper sulphate solution in the dark. Graphite electrodes were used, which were coated with paraffin except over a small rectangular space.

TABLE I.

100 g $CuSO_4.5H_2O$ per liter; graphite electrodes.
Surface of electrode = 1 sq. in. (6.3 sq. cm.)
Distance between electrodes = $2\frac{11}{16}$ inches (6.6 cm.)
Time of run = 25 minutes.

Volts	Amperes	Deposit	Temperature
1.40	0.010	No	23° C.
1.50	0.010	Yes	23°
1.40	0.010	Slight	23°
1.30	0.010	No	23°

The apparent decomposition voltage is very close to 1.40 volts because a slight deposit of copper is obtained in one experiment and none in the other. At 1.50 volts we are distinctly above the value; at 1.30 volts, distinctly below it.

A run was next made at 1.3 volts with both electrodes exposed to light. A slight deposit of copper was obtained. When the anode was screened from light a slight deposit was also obtained. Much to our surprise, a distinctly heavier deposit was obtained when the cathode was shaded and the anode was exposed to light.

Owing to the difficulty in determining the presence of slight deposits of copper on the graphite cathode, it was thought advisable to substitute a platinum cathode of the same effective area as the graphite one, the back and all but about one square inch of the face being coated with paraffin. The graphite anode was retained.

In Table II are given data obtained under the new conditions. Under the heading "Light," *C* means that the cathode was illuminated, *A* that the anode was illuminated, and *A* + *C* that both were illuminated.

TABLE II.

100 g $CuSO_4.5H_2O$ per liter; graphite anode and platinum cathode.
Surface of electrode = 1 sq. in. (6.3 sq. cm.)
Distance between electrodes = $2\frac{11}{16}$ inches (6.6 cm.)
Time of run = 15 minutes.

Volts	Amperes	Deposit	Light	Temperature
1.0	0.008	No	No	28° C.
1.3	0.010	Yes	No	28
1.0	0.008	Yes	A	28
1.0	0.008	No	No	20.5
1.0	0.008	Yes	A	23
1.0	0.008	Yes	A	25
1.0	0.008	No	No	25
1.0	0.008	No	C	27
1.0	0.008	No	A + C	28
2.0	0.040	Yes	No	19
2.0	0.040	Yes	C	22
1.5	0.015	Yes	C	22.5
1.3	0.010	No	C	28
1.3	0.010	Yes	No	25
1.4	0.012	Yes	C	26
1.0	0.008	Yes	A	27
1.0	0.008	Yes	A	27
1.2	0.008	Yes	No	27
1.3	0.010	Slight	C	20

An examination of Table II shows that we may ignore the temperature fluctuations so far as these measurements are concerned. The only place where there is the slightest trace of discrepancy is that at 1.3 volts, with the cathode illuminated; we get no deposit at 25°, and a very slight one at 20°. A precisely similar result was obtained in Table I when there was no change in temperature.

If we study the experiments without light we find that there was no deposit of copper at 1.0 volt, and that copper was deposited at 1.2 volts, at 1.3 volts and at 2.0 volts. The decomposition voltage as thus measured is therefore at least as low as 1.2 volts. Later experiments indicate that it is probably 1.1 volts. Either figure is distinctly lower than the value obtained with a graphite cathode. No experiments have been made to determine to what this difference is due.

When the anode alone is illuminated, we get a deposit of copper at 1.0 volt and at all higher voltages. Therefore we conclude

that exposing a graphite anode to the light of a mercury vapor lamp lowers the decomposition voltage of a copper sulphate solution.

When the cathode is illuminated there is no deposit at 1.0 volt, and none, or only a very slight one, at 1.3 volts. At 1.4 volts we get a distinct deposit of copper. Exposure of a platinum cathode to light therefore increases the decomposition voltage of a copper sulphate solution. A necessary consequence of these two conclusions is that a higher voltage will be necessary to deposit copper when both electrodes are illuminated than when the anode alone is. This has been confirmed experimentally, for no copper was obtained at 1.0 volt when both electrodes were exposed to light.

The results given in Table II are entirely consistent, but some later experiments were quite discordant, as may be seen from Table III.

TABLE III.

100 g $CuSO_4.5H_2O$ per liter; graphite anode and platinum cathode.
Surface of electrode = 1 sq. in. (6.3 sq. cm.)
Distance between electrodes = $2\frac{11}{16}$ inches (6.6 cm.)
Time of run = 15 minutes.

Volts	Amperes	Deposit	Light	Temperature
1.0	0.008	No	No	22°
1.0	0.008	Slight	A	22
1.0	0.008	Slight	A	22
1.1	0.008	No*	C	22
0.8	0.008	No	No	23
0.8	0.008	No	A	23
1.0	0.008	No	A	23
1.1	0.008	Yes	A	23
1.0	0.008	Yes	No	25
0.9	0.008	Yes	No	25

* In this experiment no copper was deposited on the illuminated face of the cathode but some copper was deposited on the shaded back of the cathode on a spot where the paraffin had been knocked off accidentally.

The last two experiments in Table III are quite abnormal, because copper was deposited in the dark at 1.0 volt and at 0.9 volt. Three days later copper could be deposited at 0.7 volt without the aid of light. It was thought that perhaps copper had

been deposited on the graphite electrode during the time which had elapsed since the last run and that this might be due to the action of sunlight, because the apparatus had been moved from a relatively dark corner to a place near the window. This did not seem impossible, because it is known that charcoal precipitates metallic silver from a silver nitrate solution in the dark.

If copper or some other depolarizer is formed in the pores of the graphite anode it ought to be possible to remove this unknown substance by anodic polarization of the graphite. In other words, a moderate current should "flush out" the graphite, after which no copper should be obtained at 1.0 volt. To test this hypothesis the graphite was exposed to light for twenty minutes. The light was then turned off and a deposit of copper was obtained at 0.8 volt. Owing to press of other work no further experiments were made on this day. Two days later copper could be obtained at 0.95 volt with no light. The anode was "flushed out" and then tested. At 0.9 volt no copper was obtained, but a slight deposit was obtained at 1.0 volt. The circuit was broken and the graphite anode exposed to light for twenty minutes. The light was cut off and the electrode tested at once. Copper was deposited at 0.42 volt. The anode was "flushed out" and no copper was obtained at 0.9 volt.

It is thus evident that some depolarizer is formed in or on the graphite slab and that this depolarizer can be removed by electrolysis. Experiments were now made to find out whether light was essential to this reaction. It was found that similar results could be obtained in the dark, provided the graphite was left long enough in the solution. Light is therefore not necessary to this reaction although it accelerates it.

In none of the experiments made as yet has the decomposition voltage gone below 0.4 volt, which would rather imply that metallic copper is not formed. It is more probable that a reduction takes place to a cuprous salt. This should be tested with charcoal rather than with graphite, so as to get the maximum effect. It has not been possible to do this as yet.

Since the graphite anode introduced these special variations a few experiments were made with platinum anode and cathode. The data are given in Table IV.

TABLE IV.

100 g $CuSO_4.5H_2O$ per liter; platinum electrodes.
Surface of electrode = 1 sq. in (6.3 sq. cm.)
Distance between electrodes = $2\frac{11}{16}$ inches (6.6 cm.)
Time of run = 15 minutes.

Volts	Deposit	Light	Volts	Deposit	Light
1.42	Yes	No	1.36	Yes	No
1.40	Yes	No	1.38	No	C
1.38	Yes	No	1.38	Slight	C
1.36	Yes	No	1.38	Slight	C
1.34	Yes	No	1.32	No	A
1.32	No	No	1.32	No	A
1.36	No	C	1.34	Yes	A

From Table IV we see that copper is deposited in the dark at a minimum voltage of 1.34 volts. Illuminating the platinum anode has no measurable effect, while illuminating the cathode raises the necessary minimum voltage to 1.38 volts. This result was tested qualitatively in different ways. On removing the paraffin from the back of the cathode and regulating the voltage suitably it was possible to make copper precipitate on the shaded back of the cathode and not on the illuminated front, the difference in voltage more than compensating for the extra drop of potential through the solution.[2] In another set of experiments the back of the platinum cathode was paraffined as before and one-half the front was shaded. Copper was deposited on the shaded portion and not on the half exposed to light. The screen was then moved so as to reverse the illumination, whereupon copper dissolved from the portion which had been shaded and was now exposed to light, while copper precipitated on the other half of the cathode which was now shaded. These experiments seem to eliminate any possibility of the effect being due to heating or anything of that sort.

Since a copper sulphate solution absorbs a good deal of light, the light which is absorbed must make the solution less stable and should therefore decrease the decomposition voltage. This was the result which these experiments were expected to show. As a matter of fact, the reverse is true, so some factor has been

[2] An accidental case of this sort was given in Table III.

overlooked in the deduction. The forgotten factor is evidently the electrode. It is only the solution in immediate contact with the electrode which counts so far as the electromotive force is concerned. In these experiments both the solution and the platinum electrode are exposed to light. The light causes the solution to become less stable and consequently decreases the decomposition voltage. The light also causes the precipitated copper to be less stable and consequently increases the decomposition voltage. What we measure is the difference between these two. Since the light, which is effective in tending to decompose the solution, is most intense close to the lamp and least intense at the electrode, the conditions are ideally bad for getting a maximum effect with the solution. Since copper absorbs light more completely than the solution, the chances are in favor of the effect of light on copper being greater than the effect of light on the solution. It is probable that one could vary these relative effects by using suitable monochromatic light, and it is also possible that the mercury lamp is not the best source of light for these particular experiments. It was used because it was the one available source of light which could be placed easily in the solution between the electrodes.

One experiment was tried with a graphite anode and a platinum cathode, using an electric arc so placed that the light was nearly parallel to the surface of the electrodes, the idea being that the light would only graze the surface of the electrode and that consequently one would get the maximum effect due to the solution and the minimum effect due to the electrode. This experiment seemed to give a slight positive result, some copper being obtained at 1.0 volt. It is a question whether this was a real effect of light or whether it was due to the graphite anode. The experiment would have to be repeated a number of times with platinum electrodes before I should be willing to be positive as to the results.

The fact that what one usually measures is the difference of the effect of light on the solution and on the electrode may perhaps account for the surprisingly small electromotive forces observed in special cases.

The general results of this paper are:

1. The decomposition voltage of a copper sulphate solution

between platinum electrodes is not affected appreciably when the anode is illuminated by a mercury vapor lamp.

2. The decomposition voltage of a copper sulphate solution between platinum electrodes is increased when the cathode is illuminated by a mercury vapor lamp.

3. The light tends to make the solution and the deposited copper less stable. The latter effect is the greater under the conditions of the experiment.

4. It is possible to regulate the voltage so as to make copper precipitate on the shaded portions of the platinum cathode and not on the illuminated portion.

5. Graphite absorbs something from a copper sulphate solution, presumably a cuprous salt, which acts as an anodic depolarizer and can be removed by electrolytic oxidation. The reaction is accelerated by light.

6. Owing to this adsorption the decomposition voltage for a copper sulphate solution with a graphite anode and a platinum cathode can be brought down temporarily to about 0.4 volt.

This work was suggested by Professor Bancroft, and has been carried on under his supervision.

Cornell University.

DISCUSSION.

E. P. Schoch: The effect of light upon the changes that take place at electrodes is immensely important, but it has been impossible or, rather, useless to investigate it because the differences in electromotive force produced thereby are less than the contact electromotive forces between the liquids, and so far there is no way of making these liquid contacts reproducible. When we have succeeded in finding a way of making a perfectly reproducible contact E. M. F. between electrolytes, or in eliminating the effect of the liquid contact in electrode potential measurements, these things will come to the fore with an importance that can only be guessed at, and we will reap a harvest of valuable scientific results. In the meantime it is interesting to have this brought to our attention as well as this paper brings it.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

THE TRANSFORMATION OF RADIANT INTO CHEMICAL ENERGY

By S. C. Lind.

In a previous paper[1] before this Society the author presented some evidence in support of the existence, in gas reactions taking place under the ionizing influence of certain forms of radiant energy, of an electrochemical equivalence bearing a close relation to Faraday's laws of chemical action in electrolysis. This evidence has since been materially added to and a complete report[2] was recently published showing statistically for eight different gas reactions and for three reactions in liquid systems that the actual number of pairs of ions (N), produced under the influence of α rays, is approximately equal to the number of molecules (M) entering into reaction. Proceeding from this experimentally established principle an attempt was made to explain the departure from Faraday's laws which has always been observed when the amount of chemical action produced by electrical discharge through gases is compared with the current actually passing. The discrepancy is in the direction of too much chemical action for the observed current and amounts, in some cases, to more than a thousand fold, *i. e.*, instead of 96,500 coulombs per one chemical equivalent, less than 100 coulombs have appeared to suffice in some cases.

It was proposed by the author at the Boston Meeting in April, 1912 (*loc. cit.*) that this apparently excessive amount of chemical action could be accounted for on the assumption that a large number of ions produced by electronic shock recombine in great part before reaching the electrode, in such a way as to produce their equivalent quantity of chemical action while contributing nothing to the conduction of the electric current. Accordingly

[1] Trans. Amer. Electrochem. Soc., **21**, 177-84 (1912).
[2] Journ. Phys. Chem., **16**, 564-613 (1912).

the suggestion was made that Warburg's[3] results on ozonisation in oxygen by electrical discharge could thus be accounted for, which surmise has since been confirmed in part by the experiments of Krueger and Moeller,[4] who have measured the ionisation and the ozonisation produced by electrons from a Tesla tube, and have found the same equivalence as the author had previously observed in the case of ozonisation by α rays.[5] Of course their experimental conditions were very different from those of Warburg, but their experiments at least prove that electrons of high velocity do produce ozone, and that the number of ozone molecules is equal to the number of pairs of ions. With this piece of information it ought to be possible theoretically to employ Townsend's[6] equation for ionisation by shock, to calculate the amount of ozone formed by electrical discharge; but practically this undertaking does not appear feasible with the existing experiments on ozonisation on account of the lack of the accurate data required as to the dimensions of the electrodes and their distances apart.

It seems to be a question of convention and convenience whether one should refer to this equivalence of ionisation and chemical action as illustrating the "applicability of Faraday's laws to gases."[7] Faraday's laws refer, in strict sense, to chemical action produced by passage of current and would not apply directly to that produced by recombination of ions playing no part in the conduction. For example, in the case of chemical action produced by electrical discharge in gases, there would be room for much confusion in speaking of the applicability of Faraday's laws, and it appears preferable to the author, to refer, in the case of gases, to the ionic-chemical equivalence.

At the conclusion of a comprehensive review of the chemical reactions brought about by silent discharge in gases, Warburg[8] undertook to make an *a priori* calculation of the amount of ozone that could be formed by one gram of radium per hour, assuming

[3] Warburg, Sitzber. Berlin Acad., p. 1011 (1903); *ibid.* p. 1228 (1904); Ann. d. Physik., **20**, 734-42 (1906); *ibid.* **20**, 751 *et seq.*

[4] Krueger and Moeller, Phys. Zeit., **13**, 1040-3; **13**, 729; Nernst Festsch., 240-51. M. Moeller, Diss. Danzig, 1912.

[5] Lind, Monatshefte, **32**, 295-310; Amer. Chem. Journ., **47**, 397-415 (1912).

[6] J. S. Townsend "Theory of Ionization of Gases by Collision," Constable, London, 1910.

[7] *cf.* Jellinck, "Kinetik der homogenen und heterogenen Gasreaktionen," Hirzel, Leipzig, 1913.

[8] Warburg, Jahrb. der Radioaktiv. u. Elektronik, **6**, 181-229 (1909).

ozonisation by radium to be an electrical process of the same nature as in silent discharge. This calculation was made sometime before the author investigated experimentally ozonisation under radio-active influences (*loc. cit.*), but it will illustrate the principles now under consideration to examine Warburg's premises and to correct his calculations in the light of present information as to the nature of radio-active ozone formation.

Warburg's main premises were two in number, both of which have proved ill founded: (1) that the electrical quantities concerned were the charges carried by the α and β rays; (2) that the number of coulombs associated with the formation of an equivalent of ozone by radium was the same as he found under certain conditions in the case of silent discharge. Calculating then on the basis of 1,150 grams of ozone per ampere-hour, Warburg estimated $66.5.10^{-3}$ grams of ozone per hour per gram of radium in equilibrium.

The first of the above premises is far from the truth, for as the author[9] has since shown, it is not the charges carried by the rays themselves which result in chemical action, but the charges produced by them in their flight, a number superior to the charges of the rays producing them by some 200,000 fold, which renders Warburg's estimation, with respect to his first assumption, far too low. The second of Warburg's assumptions is also not borne out by fact; instead of a very low number of coulombs per equivalent of ozone, the normal number is really required, as shown both by the author's results and those of Krueger and Moeller (*loc. cit.*). The second error influences Warburg's calculated result in the opposite direction from the first, but not to a degree sufficient for complete compensation, so that his value of $66.5.10^{-3}$ grams is considerably lower than the one I calculate, 0.72 gram per hour per gram of radium in equilibrium, which I estimate[10] on the basis of one molecule of ozone per each pair of ions.

It may be of interest to note that although both of Warburg's

[9] Journ. Phys. Chem., **16**, 606 (1912).

[10] NOTE.—It is not yet quite certain whether it requires one or two pairs of ions to form a molecule of ozone. Krueger and Moeller find *one* for Lenard rays, as the author thought to have found for α rays, but later discovered it really to have been *two* pairs in the case of highest efficiency (*cf.* Correction in Amer. Chem. Journ., May, 1913). Electrolysis also requires two pairs. It should also be mentioned that this theoretical maximum of ozone, 0.72 gram per gram-hour of radium, could never be realized, because no experimental arrangement will allow complete absorption of the rays by the gaseous oxygen to be acted on.

premises have found to be in error; in principle, the same mistake is involved, namely, that *it is the ionisation produced by the radiant energy which must be considered, and not the number of electrons reaching the electrode in the discharge tube, on the one hand, nor the number of charges emitted by the radium, on the other.* Were it not for the great difference in the mass associated with charge in the case of electrons and α rays Warburg's two false assumptions would almost have compensated each other so as to have given him a correct result.

EFFICIENCY FACTOR IN THE TRANSFORMATION OF RADIANT ENERGY.

In the foregoing, attention has been directed solely to the question of electrochemical equivalence in gas reactions, as an indication of the way in which the reactions take place. But another question of equal importance presents itself here, just as in electrochemistry, as to the efficiency factor for the transformation of radiant into chemical energy. Up to within a few years but little progress had been made in this direction. Particularly in the field of photochemistry has this problem proved unusually baffling, mainly on account of the difficulty in determining the quantity of light energy absorbed in a given gaseous system. Only recently have Warburg,[11] Weigert[12] and others been able to present some experimental data. As Warburg has pointed out, one of the chief characteristics of photochemical energy transformations seems to be their low order of efficiency, which he has sought to explain by the assumption of an intermediate reaction, possibly the splitting of molecules directly into the atoms, a process that would require much more energy than the finally resulting reaction, were it entirely molecular in nature. As we shall see presently, radio-active evidence also suggests an intermediate reaction requiring a large amount of energy and shows a correspondingly small efficiency factor, but it appears more logical to assume here that it is gaseous *ions* which constitute the intermediate products rather than atoms.

The calculations of the efficiency factor for reactions taking place under radioactive influences have hitherto been based on the heat effect of the radioactive material used and were neces-

[11] Sitzb. d. Kgl. Preuss. Akad. der Wiss., Berlin, 746-54 (1911).
[12] Zeit. f. wiss. Photographie, **11**, 381-93 (1912).

sarily limited to cases in which it was known, or at least assumed, that total absorption of the radiations took place within the system (necessarily a liquid one) in which the chemical reactions were taking place, which limitation has reduced the number of reactions for which the percentage of efficiency has been thus determined to two, the decomposition of water and of hydrogen bromide. In the former, different determinations have varied from 1 percent to 6 percent, while for the latter the author found something over 3 percent.

Another method[13] applicable to a larger number of reactions and not confined to liquids alone, will be now developed by means of the calculated ionisation and chemical efficiency of the ions previously determined by the author.[14] Let i be the radiant energy expended in the production of a pair of ions, which Rutherford[15] has shown to be $5.5.10^{-11}$ ergs for air, and which does not differ very much for other gases. Let a $(=N/M)$ be the number of pairs of ions associated with the chemical change of a single molecule in any reaction under consideration; let q be the corresponding heat of reaction, in the ordinary sense. $a.i$ is then the radiant energy expended on one molecule undergoing change, and since q is the resultant heat of reaction stored up, $q/a.i = R$, the efficiency factor for transformation of radiant into chemical energy sought. Or, taking $6.0.10^{23}$ as the number of molecules in a gram-molecule of a gas under standard conditions, we may for convenience use Q and I for q and $i \times 6.0.10^{23}$ respectively, the equation then becoming: $R = Q/a.I$.

We shall use the values of a calculated by the author[16] from data of various sources by means of the method of "average path." The values of Q are those of Thomsen. The following table (I) gives, in the first column, the reaction under consideration; in the second, the values of a $(=N/M)$ just referred to; in the third, for sake of comparison, the number of electrical equivalents that would be required by Faraday's law to bring about the same reaction electrolytically; in the fourth, the values

[13] NOTE.—This method must lead to the same results as the first one, since both depend on the kinetic energy of the radiations, which Rutherford has shown to account fully for the observed heat effects. (*cf.* Rutherford's "Radioactive Substances and their Transformations," p. 577, Camb. Press, 1913).

[14] Journ. Phys. Chem., **16**, 589 (1912).

[15] "Radioactive Substances and their Transformations," pp. 158-9.

[16] Lind, Journ. Phys. Chem., **16**, 589; Le Radium, **9**, 426-31.

of Q in Cal., taken from Landolt and Börnstein's Tabellen; and in the last, the values of R, the coefficient of efficiency, as calculated from the equation: $R=Q/a.I$.

TABLE I.

Efficiency Factor of the Transformation of Radiant into Chemical Energy.

Reaction	a	a'	Q in Calories	R = Percent Efficiency
$H_2O = H_2 + \frac{1}{2}O_2$	2.9	(2)	68.36	2.7
$CO_2 = CO + \frac{1}{2}O_2$	2.86	(2)	67.7	2.6
$O_2 + \frac{1}{2}O_2 = O_3$	2.0	(2)	34.1	2.0
$HCl = \frac{1}{2}H_2 + \frac{1}{2}Cl_2$	1.4	(1)	22.0	1.8
$NH_3 = \frac{1}{2}N_2 + 3/2\,H_2$	2.7	(3)	11.9	0.5
$HBr = \frac{1}{2}H_2 + \frac{1}{2}Br_2$	0.45	(1)	8.4	2.2

Since no great accuracy can yet be claimed for the values of a, the same degree of uncertainty must attach to R; the numbers suffice, however, to show that for those reactions brought about under the influence of α rays, the same low efficiency appears as has been observed for some photochemical reactions and also for the decomposition of ammonia by silent discharge (0.75 percent).[17] Regarding the experimental values of a it will be observed that they approach with surprising closeness to those of a' for ordinary electrolytic action, and although there is no reason to think that they should be identical with them, as has been pointed out by the author,[18] it begins to appear that they may ultimately prove to be so. The decomposition of water has also been recently studied by Duane and Scheuer,[19] from whose results the value of a appears to be about 1.5, and who calculate a value for R of about 6 percent.

Let us proceed to find what conclusions may be drawn from the above estimated efficiency factors. It may perhaps now be taken as granted that ions constitute the primary agents of reaction under the influence of α rays, and that the value of $a(=N/M)$ is usually of the order of 1 to 3. Since the energy necessary to form a pair of ions ($5.5.10^{-11}$ ergs) is larger than

[17] Pohl, Ann. d. Physik, **21**, 879-900 (1906).
[18] Lind, Journ. Phys. Chem., **16**, 590-1 (1912).
[19] Duane and Scheuer, Le Radium, **10**, 33-46 (1913).

the chemical energy stored up as final result of chemical reaction (for a reaction where $Q = 100$ Cal., q would be 6.10^{-12} ergs), it follows that the efficiency of energy transformation through the intermediation of ionisation must be low. For example, taking an ideal case, where $a = 1$ and $Q = 100$ Cal., R would then be about 10 percent; but in all actual cases, larger values of a and smaller values of Q both contribute to reduce R still further, usually to 2 to 3 percent or less, as seen in Table I. Can it then be assumed that those chemical reactions produced by other forms of radiant energy showing this same low grade of efficiency are also brought about through intermediate ionisation? This hypothesis would be similar to Warburg's except that he assumed the intermediate products to be *atoms*. However, it does not appear advisable to attempt to force the ionisation hypothesis too far in this direction. In photochemistry it is believed that the action of light is more in the nature of a resonance effect, the efficiency of which sometimes appears much greater than we have seen to be characteristic of the actions brought about by ionisation. A statistical comparison of ionisation and photochemical action, particularly in the case of ultraviolet light, seems highly desirable but presents many practical difficulties.

Hitherto only endothermic reactions have been under consideration, and, in reality, it is only among those reactions requiring an expenditure of external energy for their accomplishment that one can properly refer to an efficiency factor. But a number of exothermic reactions which do not proceed at ordinary temperature of their own free energy, are also brought about by radioactive agency. The fact that the exothermic reactions show about the same N/M ratio as the endothermic ones has already been interpreted by the author[20] as meaning that they are not to be regarded as *catalytically* influenced, and that their own free energy plays only a secondary rôle, if any, in their accomplishment. It may render this clearer to present a table similar to Table I in which R will again represent the ratio of chemical to radiant energy, but it is to be remembered that it is no longer a transformation factor, because both kinds of energy are here acting in the same direction.

[20] Journ. Phys. Chem., **16**, 611 and 613.

Table II shows that *in maximo* the ratio of $\frac{\text{chemical energy}}{\text{radiant energy}}$ reaches only 10 percent and in the other three reactions is of the same order as in Table I, for endothermic reactions, thus bearing out the claim that the chemical free energy does not materially assist the reaction in taking place under ionizing influences. This is also supported by the fact that both types of reaction follow the same kinetic equation,[21] a first order reaction falling off at the same rate as the radioactivity decays, in the case of radium emanation; *first order,* however, *not on account of any first order chemical reaction that is taking place, but because the absorption of the radiant energy is proportional to the first power of the concentration in the gaseous system,* which makes the reaction *appear as first order,* but the real reactions taking place are *immediate, not time reactions* at all, and therefore of the *zero order,* in the kinetic sense.

TABLE II.

Reaction	a	Q in Calories	R
$H_2 + \frac{1}{2}O_2 = H_2O$.....	0.8	68.36	9.8%
$2CO = CO_2 + C$.....	1.16	39.0	3.9%
$\frac{1}{2}N_2 + 3/2\,H_2 = NH_3$....	2.12	11.9	0.65%
$\frac{1}{2}H_2 + \frac{1}{2}Br_2 = HBr$.....	2.0	8.4	0.5%

SUMMARY.

1. Much experimental evidence is now at hand in support of the principle of electrochemical equivalence in gas reactions taking place under external ionizing agencies.

2. The use of the term "applicability of Faraday's law to gas reactions" may lead to confusion, especially in the case of reactions produced by electrical discharge, in which the measured current does not correspond directly to the quantity of chemical change. The term ionic-chemical equivalence in gas reactions seems preferable.

3. The theoretical maximum amount of ozone producible per hour by one gram of radium in equilibrium is estimated to be about 0.72 gm. Warburg's premises by means of which he esti-

[21] *Loc. cit.,* pp. 594-6.

mated a lower value are discussed and the sources of error pointed out.

4. A method is shown by which the coefficient of the transformation of (ionizing) radiant energy into chemical energy may be calculated from the N/M ratio.

5. For several *endothermic* reactions the transformation factor (R) is shown to have the low average value of about 2 percent, the same as has been found for some reactions produced under other forms of radiant energy.

7. The ratio of chemical to radiant energy in *exothermic* reactions is of the same low order, indicating that the influence of the free chemical energy in these reactions when produced by ionizing agencies, is very subordinate in importance, if it exists at all.

Laboratory of Physical Chemistry,
University of Michigan,
July, 1913.

DISCUSSION.

F. C. Frary: Is the phenomenon discussed the same as the electronic shock mentioned by Haber, which gives an unusually large yield of nitric oxide?

S. C. Lind: I should say it is; ionization by electronic shock occurs whenever the voltage under which the electrons travel exceeds a certain value. The necessary voltage at atmospheric pressure is very high; it requires 30,000 volts per centimeter to produce ionization, and as you increase the field above that voltage the ionization goes up very rapidly, as the new ions also produce others.

F. C. Frary: As I remember it, Haber used a low pressure and a comparatively low voltage, not over 4,000 or 5,000 volts, and had a path of 20 centimeters long, and that is why I wondered if it was possible to have the same electronic shock as you would get in the case of the ozone apparatus.

S. C. LIND: Haber and Koenig employed a high-tension arc which would further favor ionization by its high temperature.

W. R. CHEDSEY: Is the ozone produced by the Warburg process due to ultra-violet light or to electrolysis in the gas?

S. C. LIND: I have not worked under Warburg's conditions at all, but should be inclined to say that the ultra-violet light has only a secondary effect, if any. We know that ozone is produced by ultra-violet light, but I should prefer to ascribe its formation in the Warburg ozonizer to ionization of the oxygen.

A MEMBER: Is the effect the same as that of electrolysis?

S. C. LIND: Ultra-violet light does produce ionization, but the ratio of ozonization to ionization under these conditions has not yet been investigated, and I think it is very desirable for some one to undertake it, although it might prove very difficult experimentally.

E. P. SCHOCH: With reference to the two systems, those in Table I and Table II, I notice Dr. Lind differentiates between the two, in that one is a system in which energy is absorbed, and in the other energy given out. Now, in the decomposition of hydrobromic acid, for example, you may start with pure hydrobromic acid, in which case you would not be starting with a system at equilibrium, but with an exothermic system that would react with the evolution of energy. I would like to find out what the system was under which you worked in Table I.

S. C. LIND: The system in that case was liquid hydrogen bromide under its own vapor pressure. I do not recall the exact equilibrium, but it would undoubtedly be much in favor of HBr.

E. P. SCHOCH: The same would hold for the decomposition of ammonia?

S. C. LIND: Yes, I think so, unless the extent of reaction were so little that it remained on the other side of the equilibrium, which was not the case. In all the reactions of decomposition cited the extent of reaction was sufficient to carry the system far beyond equilibrium at ordinary temperature, so that they could be properly regarded as endothermic reactions on the whole, although your criticism would apply in the very early stage of the reactions.

E. P. SCHOCH: I did not mean to make the point that the reactions would not take place even if they were endothermic. In the case of ammonia I note your efficiency is 0.5 percent here, and in the reverse reaction 0.65 percent, and so they both might be endothermic and thus account for the similar efficiency.

S. C. LIND: Under silent discharge Pohl also found 0.75 percent of the electrical energy used in the decomposition of ammonia.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

PROGRESS IN ELECTROSTATIC ORE DRESSING

By Frank S. MacGregor.

The art and principles of electrostatic ore dressing have been described in several papers, as have also some of its field applications, and gradually the process has become recognized as a standard method of ore treatment.[1]

The permanent field success of the electrostatic process (generally known as the "Huff process"), commenced in 1908, with the installation by the American Zinc Ore Separating Company, of a custom separating plant in the Wisconsin zinc field, separating marcasite from the blende of that region; marcasite being a good electrical conductor, blende a very poor conductor of electricity.

The success then obtained, and which was continued, has been due to three factors:

(1) Use of electricity produced by commercial electro-magnetic generators and transformers, whose output and potential is steady and capable of easy regulation in the place of the very erratic frictional static machines.

(2) Use of concentrated and powerful electrostatic field by means of two adjacent electrodes, giving steadiness and strength to the action.

(3) Use of a grounded machine constructed almost entirely of metal, and therefore free from electrical disturbances.

The first Huff electrostatic plant in this western country was put in operation at Midvale, Utah, near Salt Lake City, for the United States Smelting, Refining and Mining Company. This plant has been in continuous operation seven days per week since

[1] Engineering and Mining Journal, July 2, 1910; T. A. E. S., October, 1910, Meeting; Electrical World, October 6, 1910; Metallurgical and Chemical Engineering, November, 1910; Mining World, December 3, 1910; Mining and Scientific Press, June 3, 1911; Engineering and Mining Journal, December 2, 1911; Mining and Scientific Press, 567; T. A. I. M. E., February, 1912, Meeting.

1909, saving 5,000,000 to 7,000,000 pounds of zinc per year, which heretofore had gone to waste. This plant now treats about 50 tons per day of zinc-iron middlings, partly furnished by the Company's concentrator, partly from custom ore from the district. The work has been very uniform since the beginning. The "feed" to the mill assays approximately 0.04 oz. Au; 2.5 oz. Ag; 2.5 percent Pb; 1.00 percent Cu; 23 percent Fe; 28 percent Zn. The separated zinc product assays 48-50 percent zinc with 3 to 5 percent iron, and the iron product about 10 percent zinc. Most of the gold, silver, copper and lead, is found in the iron product, and this product is smelted in the Company's lead furnaces. Four sizes are made in the mill, ranging from 20 mesh down. The feed to the "fines" machines often runs more than 50 percent through a 200 mesh screen. The treatment of such very fine ore is a distinct advance in the art.

Another zinc plant, put in operation in 1911, is that of the Calumet and Sonora Mining Company, at Cananea, Mexico. This plant treats 40 to 50 tons per day of zinc-copper middlings, to finer than 200 mesh. In this plant the ore is sized through 10 and on 20 mesh, on 50, and through 50 mesh; each size is then treated on its respective units. The "feed" assays 3 oz. Ag; 7 percent Cu; 3.5 percent Pb; 15 percent Fe; 30 percent Zn.

The zinc product runs about 55 percent zinc with 5 percent iron, while the copper product carries practically all of the silver-copper—14 to 16 percent copper with only 8 to 10 percent zinc.

At the Sunnyside Mines in Eureka, Colorado, an electrostatic plant has been continuously in operation since February, 1912. The tonnage treated here is about 20 tons per day, and 90 percent of the total feed passes a 150-mesh screen. Only a scrap or oversize screen is used, no sizing being necessary. The average assays of the products for a typical month are

	Au(oz.)	Ag(oz.)	Pb(%)	Cu(%)	Zn(%)	Fe(%)
Heads	0.20	15.0	11.8		30.0	10.2
Pyrite	0.33	19.0	15.6	3.6	12.6	26.1
Zinc	0.14	5.3	3.6		41.3	3.7

Because of the high percentage of heavy gangue left in the table middlings, the zinc product is not so high grade as in some plants.

The Mary Murphy Mine at St. Elmo, Colorado, is treating

electrostatically approximately 15 tons of middlings daily, all passing a 30-mesh screen. Average assays show the following work, some of the gold occurring in the blende:

	Au(oz.)	Ag(oz.)	Pb(%)	Cu(%)	Zn(%)	Fe(%)
Heads	0.38	5.9	8.0	2.2	37.6	10.1
Pyrite	0.49	8.7	14.9	5.1	8.6	30.8
Zinc	0.19	3.6	4.2	0.4	49.3	5.0

A custom zinc plant has been built at Ouray, Colorado, by Mr. David Forrester, for handling the middlings from the various concentrating mills of that district. It is equipped to handle 20 tons per day and has been operating since the fall of 1912. Some of the ores treated come from the Atlas, Barstow, Camp Bird and Mickey Breen mines. The following results indicate the work done there.

	Au(oz.)	Ag(oz.)	Pb(%)	Cu(%)	Zn(%)	Fe(%)	Ins(%)
MILL FEED.							
Class 1	0.11	59.3	6.9	0.80	25.4	16.9	19.6
" 2	0.13	13.0	7.0	1.90	28.3	22.0	2.0
" 3	0.20	11.0	1.95	2.50	14.7	30.5	5.6
SEPARATED PRODUCTS—PYRITE.							
Class 1	0.19	103.1	10.56	1.08	7.44	28.9	20.1
" 2	0.20	15.1	9.4	2.82	11.25	33.9	2.3
" 3	0.25	13.5	2.46	3.29	4.91	38.6	2.3
BLENDE.							
Class 1	0.03	13.4	2.86	0.25	44.0	4.2	19.1
" 2	0.03	9.5	3.05	0.63	56.1	3.1	1.5
" 3	0.10	4.7	0.92	0.54	42.7	6.8	14.4

Zinc smelters pay a much better price for a high-grade zinc concentrate than for a lower one, even if the impurity is only gangue, and it is not always feasible to remove all this gangue on the ordinary wet tables without entailing too great a loss of zinc. At this plant an interesting electrochemical action has been utilized to raise the percentage of zinc in the zinc product.

Ordinarily a crystal of zinc sulphide is a non-conductor, a characteristic which enables the separation just described to be made, but, if the crystal be immersed for a short time in a very weak solution of copper sulphate, it becomes coated with a film of copper sulphide. This is an excellent conductor of electricity (speaking electrostatically) and if the particle is dried at a

moderate heat, it behaves as a conductor, and is repelled by the electric charge in the same manner as pyrite, chalcopyrite, graphite or other conducting minerals. Separations of zinc sulphide from gangue have been made in this way on a laboratory scale for some time, but the method has been utilized commercially for the first time in the Ouray plant.

The treatment is very simple; the zinc product is run into tanks, covered with a 0.5 percent solution of copper sulphate, allowed to stand for a short time, the solution then drawn off for further use, the ore then dried and on passing through the electrostatic separators a 40 percent zinc product is raised to 51 percent with only 5 percent zinc remaining in the gangue product, making a recovery of 97 percent. The consumption of copper sulphate is very small, a new solution treating several lots.

There is a very general impression that electrostatic separation is adapted only to zinc ores. As the Huff process was first worked out commercially on zinc, naturally its expansion has been most rapid in this direction.

Other fields of application are now being entered. Excellent work in the concentration of crude silver ores has been done in a 30-ton mill in Austin, Nevada. The minerals of the several ores embrace several silver compounds, together with pyrite. The gangue is quartz. The ore, crushed to 20 mesh, passes through a Ruggles-Coles cylindrical dryer, is screened to 40 mesh, and then goes to the separators.

In Australia, a Huff plant concentrates molybdenite from its gangue. The crude ore is concentrated roughly by crushing and screening, the low-grade concentrates being then raised to very high grade by electrostatic concentration.

A plant is at present being installed in the Cobalt district of Canada, to improve the low-grade concentrates, thus lowering freight and treatment charges. Remarkable recoveries of the silver minerals have been indicated in the preliminary tests.

The Canadian Bureau of Mines has installed a complete Huff electrostatic equipment in its new testing laboratory, at Ottawa, Ontario, one of the finest in existence.

Two graphite mills now employ this method for removing the mica from the graphite flake, the solution of a very vexing

problem. It is not improbable that the entire concentration of some graphite ores will be done electrostatically.

Although the method has not been applied yet to the copper field, there are in this direction some excellent possibilities, especially in the concentration of those ores which occur in heavy gangues, such as garnet, epidote, barite, etc.

A recent development of great importance, both to the process and to the industry it affects, if the present indications are borne out, is for the cleaning of coal, both anthracite and bituminous. With anthracite occurs slate and other impurities which in the finer sizes are very difficult of separation. Electrostatic separation has offered a solution for this problem, reducing the ash in the "culm" from 25 or 30 percent to 10 or 12 percent.

In bituminous coal, the problem consists in the removal of ash and sulphur. Sometimes most of the sulphur present is "organic," so associated with the carbon that it cannot be mechanically removed, but when the sulphur is present as "pyrite" or in "bone," it is readily repelled, and with its extraction comes a large reduction of the ash. Activities are just entering this field.

A typical mill flow sheet for an electrostatic plant consists of a cylindrical dryer, about 4 feet x 20 feet, made of sheet iron; bucket elevator to the top of the mill, carrying the dried ore to the screens. In some cases only a "scrap" screen is needed if the sizing required is 20 to 50 and through 50 mesh. If the various minerals will break mechanically free above 20 mesh, one or two coarse screens are necessary as 6 to 12, 12 to 20 mesh. Then the ore is taken to the separators, through which it falls by gravity, the conductors and non-conductors as separated passing directly to storage bins. Returning a small amount of middlings from each separator back into the system is beneficial in increasing the tonnage and making cleaner products. These middlings finally pass out of the system in the finished products.

The power required is small, a 3-H. P. generating outfit being sufficient to electrify a plant of from one to fifty separators. The machinery of one plant is operated by 12 actual H. P., which includes power for the dryer, elevator, screens and six separators. One attendant will care for a shift, exclusive of handling ore.

In conclusion, I would say that while the installation of electro-

static separators has not been as rapid as the exploitation of some other metallurgical processes, it has developed a unique field of its own, to which no other process is as well adapted, and the tonnage treated electrostatically is steadily increasing.

60 India Street,
Boston, Mass.

DISCUSSION.

George E. Collins: One of the electrostatic plants mentioned in the paper is operated at a mine under my charge.

When we made the original tests upon which the selection of the plant was based, we found that the results from the electromagnetic method and from the electrostatic method of separation were very similar. It seemed possible to make a somewhat higher grade of zinc by the electrostatic method, but the difference which really decided us in favor of this method was the better commercial saving of the small percentage of copper present, mostly as chalcopyrite. Chalcopyrite is, I believe, more electro-positive than pyrite, whilst, containing less iron, it is rendered less magnetic by roasting; we certainly make a better saving of the copper electrostatically than we could magnetically.

As to the relative operating costs, I suppose the cost of the roasting which would have been necessary for magnetic treatment would have been considerably higher, especially in view of the small tonnage to be handled, than that of merely drying for electrostatic treatment. This advantage, however, is practically offset by the high royalty charged by the owners of the electrostatic patents.

I can entirely confirm what Mr. MacGregor says as to the regularity of working of the electrical generating apparatus. We have had little or no trouble with it; we can regulate the voltage to within a few hundred volts, and the entire electrical apparatus causes very few delays.

We, however, find it advantageous to produce a middling, which we re-treat over the same machines. In this way we can

increase the grade of our best zinc product, sometimes running it up to over 50 percent zinc. The high-grade zinc product usually contains so little gold, silver and lead that the residues from the zinc furnace are valueless. The zinc product of lower grade, on the other hand, and that produced by re-treatment of the first middling, usually has notable residue value, and can be marketed to better advantage separately on a schedule which allows more for the gold, silver and lead. Ore from the upper levels of the mine, moreover, is partially oxidized, and makes a less clean separation than that from the lower levels.

Another question is the treatment of slime. Mr. MacGregor refers to the successful treatment of material so fine that 50 percent will pass a 200-mesh screen. Nearly all of our regular middling from the wet concentrating mill will pass an 80-mesh screen, and about one-third goes through 200-mesh. This gives no serious trouble; but we also produce in the wet mill a slime zinc middling, of which 78 percent passes a 200-mesh screen, and this we find very difficult to separate satisfactorily. We have tried mixing it in small proportion with the regular feed, and this can be done without much difficulty, as the admixture with coarser particles lessens the tendency for the slime to build itself up in festoons on the electrodes and bank up on the plates. But the grade of the products is reduced to such a degree that it is doubtful if there is any final advantage in treating the slime in this manner.

The following is an example of the separation obtained by treating the slime by itself. Treating slime alone, the capacity of the machines is reduced about 50 percent.

	Gold	Silver	Lead	Zinc	Iron	Copper
	Oz. per Ton		Wet, percent	percent	percent	percent
Slime feed	0.18	7.9	8.6	31.8	8.6	1.9
Slime zinc product	..	..	7.9	36.9	6.5	1,2
Slime iron product	0.80	10.7	10.8	12.6	31.8	2.2

Mr. MacGregor does not refer to the dust loss. It is necessary that the feed should be absolutely dry, so as to avoid moistening the air in the slightest degree. With fine, warm ore, handled by elevators and conveyors, and falling freely in this condition from shelf to shelf of the Huff machines, a great quantity of dust is necessarily produced. In order to make the temperature in the plant bearable, windows and doors are kept open, so that

considerable dust is carried off by the wind, and a layer of fine dust is observable for several acres on the lee side. An attempt to "take stock" over a period of several months indicated a loss of 3 percent of the total material entering the plant.

In drying the ore, heated air is drawn by means of a fan through a cylinder in the opposite direction to that in which the ore is passing, discharging into successive dust-chambers. From the first of these the collected dust is returned at intervals to the cylinder. In the second, only about 2 tons of material has collected so far. Of this, 92 percent passes a 200-mesh sieve. A sample assayed as follows:

Au.	Ag.	Pb.	Zn.	Cu.	Fe.
0.32	9.9	8.0	24.5	3.9	8.5

So far, we have found no way of separating this material, which is commercially of small value. If it represents the 3 percent which is blown away through windows, etc., the loss of the latter is not a very serious matter.

F. S. MacGregor: In regard to Mr. Hansen's comments (communicated in the discussion on the paper of Lyon and Kenney) regarding the operation by the same company of an electromagnetic plant, the conditions under which the electrostatic process entered the Wisconsin field were such, in a business way, that the other plant was kept in operation. It was kept in operation when the electrostatic plant was destroyed by a fire, and had nothing to do with the relative value of the processes. In regard to the non-rebuilding of the electrostatic plant, the mill had just been enlarged and remodeled when it burned, and plans were immediately made for the construction of an all-steel and concrete building. That the plant was not rebuilt was not due to the inability to separate the ore, since the plant record during its years of operation shows differently, but because this plant was the first commercial operation of electrostatic separation, and during the time of its development the difficulties that are always met with in starting up a new process and transferring it from laboratory to field operations had to be overcome before the plant was profitable. Then there was the strong competition from the magnetic plant with large financial backing. To meet the competition was quite a strain, and, as this Wisconsin ore is peculiarly

adapted to magnetic separation, it was decided to go to the Western field and utilize the experience gained in treating the complex ores of the West. This was thought rather better than to make an investment necessary for the size of plant desired for operation in Wisconsin. I am glad to explain this, because from the non-rebuilding of the plant the inference might be drawn that the process had failed to work. But the plants in operation show what can be done, and claims are not made that electrostatic separation will treat all ores.

There is a word I intended to say with respect to Mr. Collins' remarks on the gold and silver. Where the gold or silver is in the iron or lead products mechanically free, they can be easily separated, but there is a good deal of sulphide zinc ore that carries gold and silver either in the sulphide itself or in the leaves of the crystals that cannot be separated by any process other than chemical or smelting. When these gold and silver values are sent to the smelter with the zinc, part of it can be recovered in the residues, and these have to be shipped to the lead smelters.

The reason I did not mention dust loss was because its control is purely a matter of individual opinion at the various plants. It has in most cases been reduced to 1 or 2 percent, and by the use of a proper dryer or fan or suitable coverings the amount of dust allowed to escape into the mill has not been sufficient to cause any complaint from the millmen.

In regard to Mr. Hansen's remarks about California ores not being amenable to electrostatic separation, that is not entirely true, but, as far as the Bully Hill ore is concerned, while it could be separated, it is not at all practical or feasible, because it is so finely crystallized and the sulphides so intimately mixed that too fine grinding is required.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

THE CARNOTITE INDUSTRY

By Siegfried Fischer.

Since the advent of radium production from pitchblende, endeavors have been made to obtain larger quantities of ores containing this valuable element. While radio-activity is observed in many minerals, these as a rule occur only in small amounts, and, with few exceptions, are merely of scientific interest. All minerals containing uranium and thorium will, in all probability, hold one or more of the radio-active elements radium, actinium or polonium. But few of these ores are of commercial value.

A list from Rutherford's Radio-activity, showing some of these ores with their "saturation currents," will make this clearer:

Pitchblende	Uranium, 50%—80%	1.6×10^{-11} to 8.3×10^{-11}	ampere
Carnotite	Uranium, 42%—51%	6.2×10^{-11}	"
Chalcolite		5.2×10^{-11}	"
Autunite	$CaO.2UO_3.P_2O_5.8H_2O$	2.7×10^{-11}	"
Orangite	Uranium, 1%—10%	2.0×10^{-11}	"
Clevite		1.4×10^{-11}	"
Samarskite	Uranium, 8%—10%	1.1×10^{-11}	"
Aeschynite		0.7×10^{-11}	"
Monazite	Thorium	0.5×10^{-11}	"
Fergusonite	Uranium, 1%—6%	0.4×10^{-11}	"
Thorite	$ThSiO_4$	0.3×10^{-11} to 1.4×10^{-11}	"
Xenotite		0.03×10^{-11}	"

These values of the saturation current are obtained by the usual method, employing two electrodes 8 cm. in diameter and 3 cm. apart and insulated from each other. One of these electrodes is covered with the material to be tested, and the maximum current reached, when the impressed voltage is gradually increased, is measured. This "saturation current" is an indication of the radio-active strength of the mineral.

Mineralogical investigations have shown two sources which

contain uranium in sufficient amounts and of which there are large enough deposits to justify their commercial working. These are pitchblende and carnotite. While there are considerable deposits of monazite, the thorium mineral which also carries at times small amounts of uranium, its comparatively low radio-activity eliminates it from becoming a true source for radium production.

Up to recent years pitchblende was *the* commercial uranium ore. The largest deposits of pitchblende are found in Austria, especially at Joachimstahl. Other deposits are in Saxony, Cornwall and at several places in the United States. The most important one of the latter is at Central City, Colo. Further investigations will undoubtedly open up other deposits of this nature, but, as the topic of pitchblende is an old chapter in the history of radium, I will confine myself chiefly to a discussion of carnotite.

Description.—Carnotite is a dark green to canary yellow, soft, powdery impregnation in some gangue material such as sandstone, limestone or clay, and on rare occasions in coal. According to the percentage of uranium in an ore, the yellow color and the radio-activity are more or less dominant.

Absolutely pure carnotite is exceedingly hard to obtain, due to its nature and occurrence. While many chemical formulas have been given to it, none of them has been definitely accepted. However, it seems to be established that it is a vanadate of uranium and potassium. The complexity of carnotite will be shown best by quoting two complete analyses by W. F. Hillebrand:*

	Percent	Percent
UO_3	54.89	54.00
V_2O_5	18.49	18.05
P_2O_5	0.80	0.05
As_2O_3	Trace	None
Al_2O_3	0.09	0.29
Fe_2O_3	0.21	0.42
CaO	3.34	1.86
SrO	0.02	Trace
BaO	0.90	2.83
MgO	0.22	0.14
K_2O	6.52	5.46
Na_2O	0.14	0.13

* Amer. Jour. of Science, **10**, 138 (1900).

	Percent	Percent
Li_2O	Trace	Trace
H_2O, 105°	2.43	3.16
H_2O, 350°	2.11	2.21
PbO	0.13	0.07
CuO	0.15	Trace
MoO_3	0.18	0.05
SiO_2	0.15	0.20
TiO_2	0.03	?
CO_2	0.56	None
Insoluble	7.10	10.33
	98.46	99.25

A simple chemical test for the yellow and yellowish-green carnotite ores, distinguishing it from sulphur and molybdite, is to heat the ore with hydrochloric acid until nearly dry, dilute with water, filter and add a few drops of concentrated nitric acid to the filtrate, followed by a small amount of hydrogen peroxide, when a reddish-brown color will be given to the solution. This coloration is due to the vanadium. Commercial vanadium ores free from uranium are as a rule green to black in color, or red to reddish-brown. The darker carnotite ores, varying from light green to nearly black, require, besides the chemical test, radio-active examination.

Occurrence.—The main sources of carnotite, so far as we know, are Colorado and Utah. The Colorado belt encloses the following counties: Dolores, San Miguel, Montrose and Rio Blanco, where the ore is found in considerable quantities. Other deposits of smaller scope have been located in Montezuma, Delta and Mesa Counties, but these to my knowledge are not being worked. The Utah deposits are found in the eastern part of the State, adjacent to Colorado, comprising parts of Emerson and Grand Counties. The occurrence in both States is similar; the ore is found usually in sandstone, clay or limestone formations. In the neighborhood of Placerville, Colo., the material is found to a great extent, in a formation of the Triassic age, called the La Plata formation, and there the values are limited to the lower portion. The Colorado ores, as far as my experience goes, are of a richer nature, and have not the brownish tinge like those

of Utah. Some of the very low grades which I have obtained from Greenriver, Utah, are almost red in color.

I have analyzed carnotites from Wyoming, Nevada, New Mexico, South Dakota and California, and, while some of these ores made a very good showing in the laboratory, none of these deposits seems to be worked. Evidently they are not large enough to be of commercial importance.

The only fact definitely known is that carnotite is present as a secondary impregnation. Theoretical assumptions as to its origin have been made, but actual data are lacking.

Industry.—Up to recent years nearly all the carnotite was shipped to Europe. All the radium made was a foreign product, and it was thought that America was incapable of utilizing its own crude material. This idea is now a thing of the past, as only last month $30,000 worth of radium was shipped abroad by an American firm. It will probably not be long before this country is one of the main producers of this valuable element.

The intrinsic value of carnotite lies in its uranium and vanadium contents, and on these factors the commercial value is based. It is rather hard to give definite figures which will hold true in all cases, as the signed contracts between the dealer and the buyer are of an extremely varied nature. The only data that I can vouch for are from a Colorado firm selling carnotite, and the figures held for last year.

The contract stated that ores must contain at least 2 percent U_3O_8 and a minimum of 5 percent V_2O_5 if anything at all was paid for the vanadium. On this bases the U_3O_8 sold at $1.25 per pound, and the vanadium oxide at $0.30 per pound. For foreign shipments the cost per pound of V_2O_5 was $0.70 f. o. b. Hamburg or Liverpool. The price for the uranium to these ports is not known to me. For each percent over the percentage stated in the contract the price per pound increased $0.05.

The same firm claims that the profit per ton is $20, and the net cost per ton of ore is between $50 and $60, including sampling and analyzing.

This crude industry is very simple. The ore is mined and

sampled, analysis is made at both ends, and the product is sold on contract. But more recent researches performed in this country will undoubtedly put the carnotite industry on a more scientific and commercial foundation.

Much of the initial work was accomplished in the laboratories of the Colorado School of Mines. A sum of money was donated by the late Thomas Walsh for research upon rare metal work. While this work has not proven a success, some credit must be given to it, as it has instigated the interest along the lines of pitchblende and carnotite. About two years ago these funds were withdrawn from the School of Mines at Golden, Colo., but the interest in the vanadium, uranium and tungsten ores has not abated. On the contrary, much work is being done by private concerns, by educational institutions and recently by the Government.

While carnotite as a mineral has been known for quite a time, its commercial value was only appreciated since the discovery of radium. Publications along commercial lines are therefore scarce, since manufacturing concerns, as a rule, do not indulge in broadcasting their discoveries.

CONCENTRATION.

In the handling of other mined minerals the treatment usually begins with concentration. I will therefore first discuss whether or not concentration is a desirable operation in the case of carnotite. According to a few specifications with which I am acquainted, European trade demands that the material shall be delivered without mechanical change. This evidently means that the concentration takes place abroad or else that a chemical process is employed whose expense is so low that the firms handling this sort of ore can better afford to obtain larger shipments of low-grade material than smaller amounts of high-grade concentrates.

In an article published in *Metallurgical and Chemical Engineering,* June, 1912, I have treated this subject in detail, and a short outline of this article may be given here.

Considering the fact that the dealer in carnotite in most instances bears the freight expenses, that the crude ore can be reduced to one-third of its original volume, and finally that the ton value of the concentrates usually increases threefold, a concentration of the ore seems to suggest itself.

The concentration is based on the physical nature of the ore, and is purely a mechanical operation.

Ore is mined
↓
Grizzly → Lumps to crusher
↓
Belt conveyor takes fines
↓
Bins
↓
Rough, medium and fine rolls
↓
Dorr classifier of special construction.
Here concentration and separation are performed simultaneously.
↓
Concentrates
↓
Dorr slime thickener
Eliminates most of the water.
↓
Drier
↓
Storage bins
↓
Sold or extracted.

I have tried this process on dozens of samples from various localities, on both high and low-grade ores, and have found the concentration ratio, with very few exceptions, to be 3:1. To elucidate the value of a process of this type more clearly, let us assume that a shipment of 30 tons of carnotite is to be made, the crude ore running 2 percent V_2O_5 and 3 percent U_3O_8. Returns without concentration will be based on these figures. To obtain 30 tons of concentrates, 90 tons of the above ore would have to be subjected to the treatment. The returns would then be figured on a ton value running 6 percent V_2O_5 and 9 percent U_3O_8. As the cost of concentration is easily within the limits attributed to mechanical concentration, the application for practical purposes seems justified.

An item of importance in this process is the mesh to which the ore should be crushed. Its fineness should be as nearly as possible the same as that of the gangue material. If possible the final rolls should be of such construction that one of the rolls is adjustable. Practically speaking, an ideal condition cannot be obtained, as some fines will always be produced. But the amount of gangue found in the concentrates is so small that in most cases it can be neglected.

The following tests were run to prove the efficiency of the process:

1. Crude ore, concentrates and tailings were subjected to a microscopic examination.

   Results: Crude ore showed carnotite as a powdery coating. Concentrates showed mostly powdered carnotite, and at times a few gangue particles. Tailings composed of gangue with traces of carnotite.

2. Crude ore, concentrates and tailings were analyzed for uranium and vanadium.

   Results: Concentrates showed approximately three times the value of the crude ore. Tailings showed either traces or none of the values.

3. Crude ore, concentrates and tailings were put to the following test: The same amounts by volume of the individual products were put into similar covered boxes. These were placed over iron washers of the same size, resting on photographic plates enclosed in black paper. Thus prepared, the individual plates were simultaneously exposed in the dark for seventy-two hours and then developed.

   Results: Crude ore showed radio-activity. Concentrates showed the radio-activity vastly increased. Tailings showed no radio-activity.

The accompanying photographs (Fig. 1) show the results of this treatment.

There is at present no patent granted for a scheme of concentrating carnotite ores in the United States, and the European patents referring to this product are unknown to me, so that I am not in a position to compare this process with any other.

One question which has aroused some interest is whether or not this process may also be applied to carnotites in a clay gangue. It seldom happens that a man concentrating carnotite ores would be thrown up against clayey material alone. The mixing of a certain percentage of clayey material with ore containing sandstone gangue will eliminate any trouble in that direction. I have had opportunity to try this several times, and the results always proved satisfactory.

CHEMICAL EXTRACTION.

The concentrated product also has a great advantage from the extraction point of view. The amount of chemicals used may be considerably reduced, and as an initial source for further treatment it is a product of nearly ideal purity in comparison with the material handled today.

Merely as a source of vanadium, carnotite cannot be classed under the commercial ores of this element, as it is unable to compete with patronite, roscoelite and vanadiferous sandstones, and in all probability in the near future it will be unable to compete with vanadinite. The patronite deposits of South America are claimed to be limitless in their amount, and the other ores mentioned occur in greater deposits than carnotite. It is rather to its uranium and therefore radium content that carnotite owes its gradual advancement in the commercial world.

There are three stages in any procedure for the extraction of the values from the ore:

1. The extraction of the values.
2. The separation of the uranium and the vanadium.
3. The obtaining of the radium from the uranium.

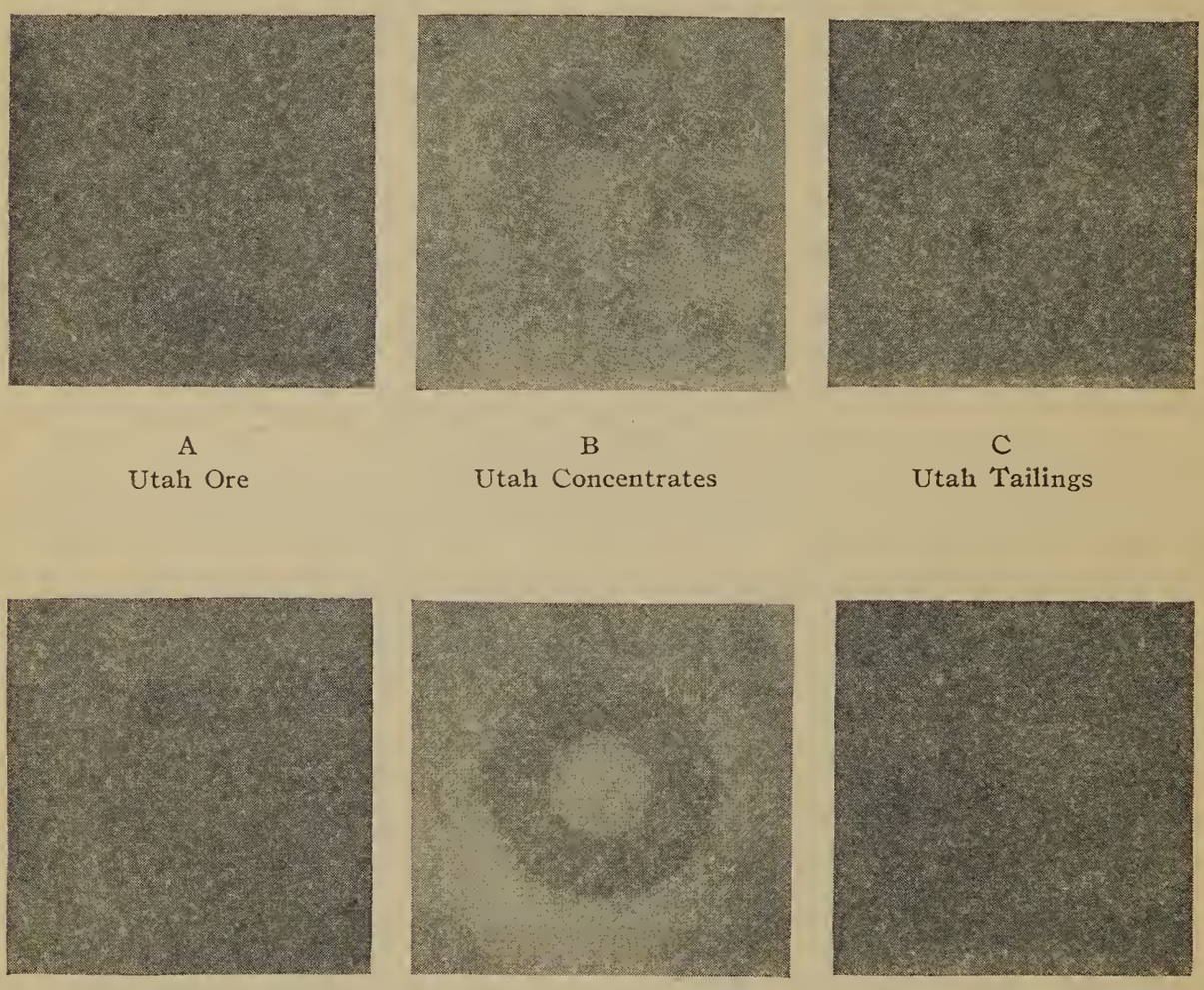
Two technical methods of treatment are at the present time in operation, the acid and the basic extraction methods.

Acid Process.—A representative example is the process of H. Fleck, W. G. Haldane and E. L. White, U. S. Patent 880,645, March 3, 1908. The outline of the process is:

Ore
↓
Crush to 20–40 mesh
↓
Agitate with 15–20 percent H_2SO_4, amount of acid used depending on nature of ore.
↓
Filter → Solution contains Va, U, Cu and other impurities.
↓
Heated, brought in contact with new ore, to neutralize solution. Here part of the Va and U is deposited on the newly treated ore, enriching it.
↓
Filter → Solution
Filter → Residue: wash with dil. H_2SO_4
↓
Filter
↓
Filter → Gangue
Filter → Filtrate
↓
Add enough H_2SO_4 to bring it to 15-20 percent and reuse.

Enriched ore → same treatment as above.
↓
Final product to → Stock
↓
Treat with sulphurous acid, obtained by conducting SO_2 gas into the stock solution. This reduces Fe and Va to "*ous*" form, at the same time converting corresponding amount of SO_2 to SO_3, which with H_2O forms H_2SO_4.
↓
Add enough ore to neutralize this H_2SO_4.
↓
Filter → Residue: ore.
Filter → Filtrate: values plus impurities.
↓
Add enough limestone to begin the precipitation of the Va, Cu and U. $CaSO_4$ is formed.
↓
Filter → To filtrate add requisite amount limestone and boil. This precipitates the values and some iron as basic sulphates and carbonates with hydrated calcium sulphate.
↓
Dried
↓ Shipped or ↓ *e. g.*, Treat wet or dry with sulphurous acid, boil SO_2 off. U precipitated as basic sulphite.
↓ Reused.
↓
Filter
↓ Residue ignited to oxide.
↓ Filtrate: precipitate Va and Fe with CaO.

On June 9, 1908, U. S. Patent 890,584 was granted to the same gentlemen, which in its essentials is the same, but provides for certain ores which are capable of being treated directly with sulphurous acid, this being furnished by roasting pyrites, and reused.



A
Utah Ore

B
Utah Concentrates

C
Utah Tailings

1
Roc Creek Ore

2
Roc Creek Concentrates

3
Roc Creek Tailings

Fig. 1.

The acid process has never appealed to me for the reason that so many impurities are brought into the extraction product, and must be gotten rid of at some stage of the operation. The patent also shows that the process is a tedious one, requiring many steps, thus increasing the time, reducing the output and increasing the cost. In spite of these facts, however, a plant is in operation based on these patents. Whether or not it is a success time alone can show, since it has been running less than a year.

Basic Process.—The principle underlying this process is essentially the same, namely, obtaining the vanadium as a soluble vanadate and the uranium in form of an insoluble uranate. These are then separately treated in order to obtain a vanadate of some metal such as Fe, Cu or Pb. The uranium may be further purified or sold as uranium residues.

To show the simplicity of the basic treatment a general outline will be given. The scheme used here is not from any one patent, but rather one embodying all patents appertaining to this subject.

Ore: Mined
↓
Crushed to suitable size
↓ either: Mixed with alkaline carbonates or alkalies and roasted
↓ or: Heated with a hot solution of alkaline carbonates or alkali.
↓
Both these methods change the vanadium to a soluble, and the uranium to an insoluble form.
↓
Leach with water
↓
Filter

Residue: contains U values and some Va. Besides these is the gangue.
↓
Extract U and rest of Va.
↓
Refine electrolytically or by some other suitable method, gaining both the U and the Va.

Filtrate: most of the Va.
↓
Neutralize
↓
Precipitate Va as Fe, Cu or Pb vanadate, filter and dry. Treat vanadate by Goldschmidt, electrolytic or electric furnace method.

The refining of the uranium residues I will discuss in another paragraph.

Most of the work along the line of the basic extraction of the values from carnotite was done by Warren F. Bleecker, now with the Standard Chemical Co. of Pennsylvania, and some of his patents of value for this industry will be outlined. Some of these are typical vanadium patents, but one of them is a decided step forward for carnotite.

U. S. Patent 1,049,330 (January 7, 1913), of Warren F.

Bleecker. The trend of this patent is to produce an insoluble vanadate which, on dissolving in acid, is subjected to electrolysis. The compound is decomposed, depositing the metal (copper, etc.), forming the vanadate as a cathode product, the vanadium remaining in the electrolyte. The solution is then evaporated and vanadic acid obtained. Some salts used in this fashion are $Cu_3(VO_4)_2$, $CuVO_3$ and CuV_4O_{11}. U. S. Patent 1,015,469 (January 23, 1912) shows how Mr. Bleecker obtains his vanadates.

It can be readily observed that the electrolytic refining of the vanadate also applies to carnotite. The neutral sodium vanadate solution is treated with a commercial copper salt to precipitate the vanadium as an insoluble copper vanadate. This is followed by filtering and electrolyzing as above mentioned.

In experimental work performed in the laboratories of the Colorado School of Mines my experience has been that Cu and Ni vanadates were best suited for electrolytic refining. Ferro-vanadate requires too much time for the complete elimination of the iron, and I have never managed to overcome this difficulty. Since my work has been cut short at that institution I am unable to give the curves showing the ratio between the time and the amount of Cu, Ni and Fe deposited, as the number of experiments completed would not justify me in making definite statements. The same holds true for the most suitable conditions under which electrolysis could be accomplished commercially. These data are undoubtedly known to some manufacturing concerns, but the knowledge is kept a secret. The conclusion drawn by me at the time was that vanadates formed from metals whose salts were easily electrolyzed were best suited for this method of treatment, and, while their production was more expensive than those of some of the other metals, the time gained justified their use.

A patent of interest to the carnotite industry is Mr. Bleecker's U. S. Patent 1,050,796 (January 21, 1913), describing an electrolytic separation of the U and Va. The electrolysis takes place in an alkaline sodium carbonate solution, the values then being present as sodium uranyl carbonate and sodium vanadate. It is as follows:

Solution containing values
↓
Heat to 90° C.
↓
Add heated sodium hydrate until uranium is precipitated as sodium uranate and uranylhydrate.
↓
Filter and wash

Precipitate U and Va.
↓
Dissolve in H_2SO_4. This forms uranyl sulphate.
↓
Add Na_2CO_3 to alkalinity
↓
Electrolyze, using Fe, Cu or Ni electrodes. The Va will then be deposited as an anode product and will be a vanadate of the metal used as electrode.
↓
Filter
↓
Filtrate U
↓
Precipitate in any of the known ways.

Filtrate Va
↓
Precipitate Va.
↓
etc.

One more patent I will allude to, as it is of value for the extraction of the values from carnotite concentrates. This is S. Fischer's U. S. Patent 1,054,102 (February 25, 1913).

The concentrates are mixed with a certain amount of caustic alkali and made up to a paste with water. This is subjected to heat, and the water maintained at approximately constant volume. After sufficient treatment the residue is dried and slightly roasted. It is then leached under friction with water, filtered and washed. The filtrate contains the Va, which is precipitated as ferro-vanadate, filtered, dried and treated by the Goldschmidt or an electric furnace process to give ferrovanadium. The residue left after the first filtration is uranium concentrates, and after careful washing will show at times some vanadium. If this be the case the electrolytic separation of the Va from the U would take care of the difficulty, but as a rule the Va left in this residue is negligible. These uranium concentrates may be shipped directly or treated for radium. The precipitation of the vanadium as Cu and Ni vanadates has been tried on several samples, and the electrolysis performed with these products gave very satisfactory results.

The final step in the carnotite industry today is the extraction of the radium from the uranium concentrates. Very little has been made public in this field. One patent has been granted in this country, No. 1,049,145, to Sidney Radcliff (December 31, 1912). According to competent authority it is of little avail in the carnotite industry, and will therefore not be discussed.

Radium being closely allied to barium, a barium separation was performed on pitchblende ores. On this basis I have treated several uranium concentrates obtained from carnotite, and have had fairly good results. The quantity of concentrates used having been comparatively small, the barium and radium sulphate residues were not of sufficient amount to make absolute examinations on them, but the uranium residue, after treatment to eliminate all foreign matter, showed a decided decrease in radio-activity. Three methods are claimed to be applicable to carnotite for the extraction of the radium, their commercial value depending greatly on the locality in which the manufacturing of the radium salts will take place. For further knowledge on this topic we will have to await the publications of the Bureau of Mines in Denver, which is doing extensive and most valuable work in this direction.

As no industry can be considered a success until all the by-products are utilized, the carnotite industry can not be classed as a fully developed commercial process. Vanadium has an established market; the demand for radium is increasing steadily, but what becomes of the uranium? When we consider 2,600,000 parts by weight of uranium will produce only one part of radium. it is evident how insignificant, commercially speaking, the amount of radium is in comparison to uranium. And yet for this important by-product of the carnotite industry there is practically no market demand. Some uranium is used in the glass and porcelain industry as a color medium, but the amount is a mere bagatelle. This subject calls decidedly for research. Some of the large steel companies have tried to use uranium in their line of work, but with little or no success. This seems to have discouraged investigators, and the work spent on this element, outside of producing radium, has nearly ceased. I would like to suggest investigating alloys of uranium with other metals besides iron. It may be that it will contribute properties to

some that may be of the greatest value and open up a market for what is now practically a waste product.

In submitting this paper I am not presenting much that is new, but simply wish to encourage investigation in this country of an industry which up to recent date has been practically restricted to Europe—the production of radium. The raw material at our disposal amply justifies such a course.

I wish to express my sincere thanks to Mr. H. C. Parmelee for his kind assistance, and to Dr. Moore, of the Government Bureau of Mines, at Denver, for some suggestions.

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Golden, Colo.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

SOME OBSERVATIONS ON BASE METAL THERMOCOUPLES

By O. L. Kowalke.

The thermocouple as a means of measuring temperatures seems to be used by a large variety of industries, if one may judge from the records of sales. It is peculiarly adapted for this purpose because of (1) the small volume the junction occupies, (2) the robust nature of the couple itself as compared to a thermometer, (3) the simplicity of the measuring or indicating instruments, (4) the rapidity with which the measurement can be made. Up to about seven years ago the platinum couple, which had been thoroughly investigated and tried out, was almost alone in the field. Since then there have appeared on the market couples made of the baser metals, such as iron, nickel-chromium, nickel, cobalt and other alloys. These couples came into favor because of their cheapness as compared to the platinum couple both as to first cost of the outfit and the cost of renewals of the couple itself, the high electromotive force generated and the use of a robust double-pivoted millivoltmeter. Among the causes for the inaccuracy of the platinum couple were heterogeneity of the wires in the couple and their contamination by metals or gases from the place where the temperatures were to be measured. All but the first of the causes named can be avoided after the couple is made, but the wire must be made homogeneous before the couple is completed.

Since the voltage generated by the couple for a given temperature is the summation of all the electromotive forces due to the contact of two dissimilar metal portions, it can readily be seen that it is imperative that the metal be as homogeneous as possible if the electromotive force indicated be that resulting from the two metals at the hot junction. If, then, the couple is composed of metal which is not homogeneous, then at each junction of two dissimilar metals there will be set up a voltage

which is a function of the temperature existing at that point. Should the position of the couple be changed with respect to its depth of immersion, then the resulting voltage will be changed even though the temperature had not changed at all. A couple made of wires which are perfectly homogeneous is apt to be rather expensive, and the only combination which seems to fulfill this condition is the platinum and platinum-rhodium couple.

It is possible to get a couple which is sufficiently homogeneous so that the indications are satisfactory for many commercial purposes where an accuracy of about 25 to 50 degrees is wanted. This condition is realized in the combinations of which base metal couples are made. Not all of the couples meet this condition with the same accuracy, and not all of the couples retain their original accuracy. Some couples appear to change or deteriorate with use more than others, due to oxidation, crystallization, segregation of the metal and other undetermined causes.

It was the purpose of this investigation to determine how electromotive forces of couples varied with the temperature, and how constant this force remained with successive heatings and coolings, and also when exposed to various temperatures for extended periods of time.

PROCEDURE.

Couples were purchased from five of the prominent makers, and were cut to lengths of about 18 inches. To each couple about three feet of flexible lamp cord was soldered, and the wires at the point of soldering and all other required points were carefully insulated from each other.

The couples under test were all compared with a platinum and platinum-rhodium couple No. 3P, which had been standardized against a similar couple, "B. S. No. 120," certified to by the Bureau of Standards. The latter couple had not been used since it came from the Bureau of Standards, and was used only to standardize couple No. 3P at the beginning and at the end of the series of tests.

Couple No. 3P changed slightly during the investigation, and a correction was made for the indications of each series of tests. It was assumed that the change was a progressive one, and the

total error divided into equal parts and the proper correction made in the corresponding chronological series.

All couples were calibrated over the range given by the manufacturers in their catalogues, and only one base metal couple at a time was compared with the platinum couple No. 3P.

Series 1. All couples were first calibrated against No. 3P with a length of about four inches heated.

Series 2. All couples were again calibrated, but with a length of about 15 inches heated to determine the effect and presence of heterogeneity in the wires.

Series 3. All the couples were now subjected at one time to a constant temperature of 400° C. for a period of about twenty hours. After this treatment each couple was again separately calibrated with a length of 15 inches heated.

Series 4. A second treatment consisted in heating all of the couples together at a constant temperature of about 600° C. for a period of twenty-four hours. After this treatment each couple was calibrated separately, the idea being to determine the effect of heat treatment at 600° C. over that at 400° C.

Series 5. All the couples were now heated together for a period of twenty-four hours at a temperature of 800° C., and afterwards separately calibrated as before.

The calibrations and heat treatments as indicated in Series 3, 4 and 5 were made with each of the base metal couples heated for 15 inches of its length.

FURNACES.

Electrically heated tube furnaces were used in all of the tests. One furnace was 10 inches long, and the other was 20 inches long, each having a tube about one inch internal diameter, and were used for the calibration at 4 inches and 15 inches depth of immersion, respectively. A tube furnace about 2½ inches in diameter and 24 inches long, so that all should be treated together, was used for the heat treatments at the temperatures of 400°, 600° and 800° C. The current on the furnace was controlled by means of a bank of incandescent lamps, and could be adjusted

so that the temperature remained stationary for an indefinite period.

METHODS OF MEASUREMENT.

All measurements were made on Leeds & Northrup Type K potentiometers, and only one base metal couple at a time was compared with the standard platinum couple, and the voltage of each couple was determined by a separate potentiometer, the standard cells for each potentiometer being checked for accuracy.

Asbestos discs slightly less than the diameter of the tube, perforated in the center, were slipped on each couple and served to keep the couples centered in the furnace. The two couples were then introduced into the furnace so that the junctions of the couples met. The temperature of the furnace was then raised to about 300° C. for the first point so that a good reading on the platinum couple could be obtained. From this point up the temperature was increased by intervals of about 100° C. At each point the temperature was maintained stationary so that constant readings on both couples were obtained for a period of about two minutes. Thus all lag in the indications of the couples was avoided. During the entire procedure the cold junctions of both couples were kept at zero degrees Centigrade by surrounding them with a bath of melting ice, care being taken to avoid a circulation of air about the cold junction.

COUPLES USED.

The base metal couples used in this investigation are designated as follows:

Couple No. 6A, composed of wires 1/8 inch square and covered throughout the length, excepting about 2 inches, with a winding of asbestos cord, over which there was applied a solution, presumably sodium silicate. This couple had for its negative element an alloy of nickel and chromium, and the positive element one of nickel and iron.

Couple No. 7A was made of No. 8 B. & S. gauge wires, the negative element being composed of nickel with a small amount of aluminum, and the other element a nickel-iron alloy. This couple was purchased with both wires bare, and for the purpose of test an asbestos sleeve was drawn over one of the wires.

Couple No. 18 was composed of two No. 10 B. & S. gauge wires, each of which was wrapped with asbestos thread and painted over with carborundum paint. The negative element was made of nickel with a small amount of aluminum, and the positive element of iron with a small amount of nickel.

Couple No. 19, composed of two No. 10 B. & S. gauge wires, one of which was covered with an asbestos sleeve over which there was a coating of carborundum paint. The negative element consisted of nickel containing a small amount of iron, and the positive element contained iron with a small amount of nickel.

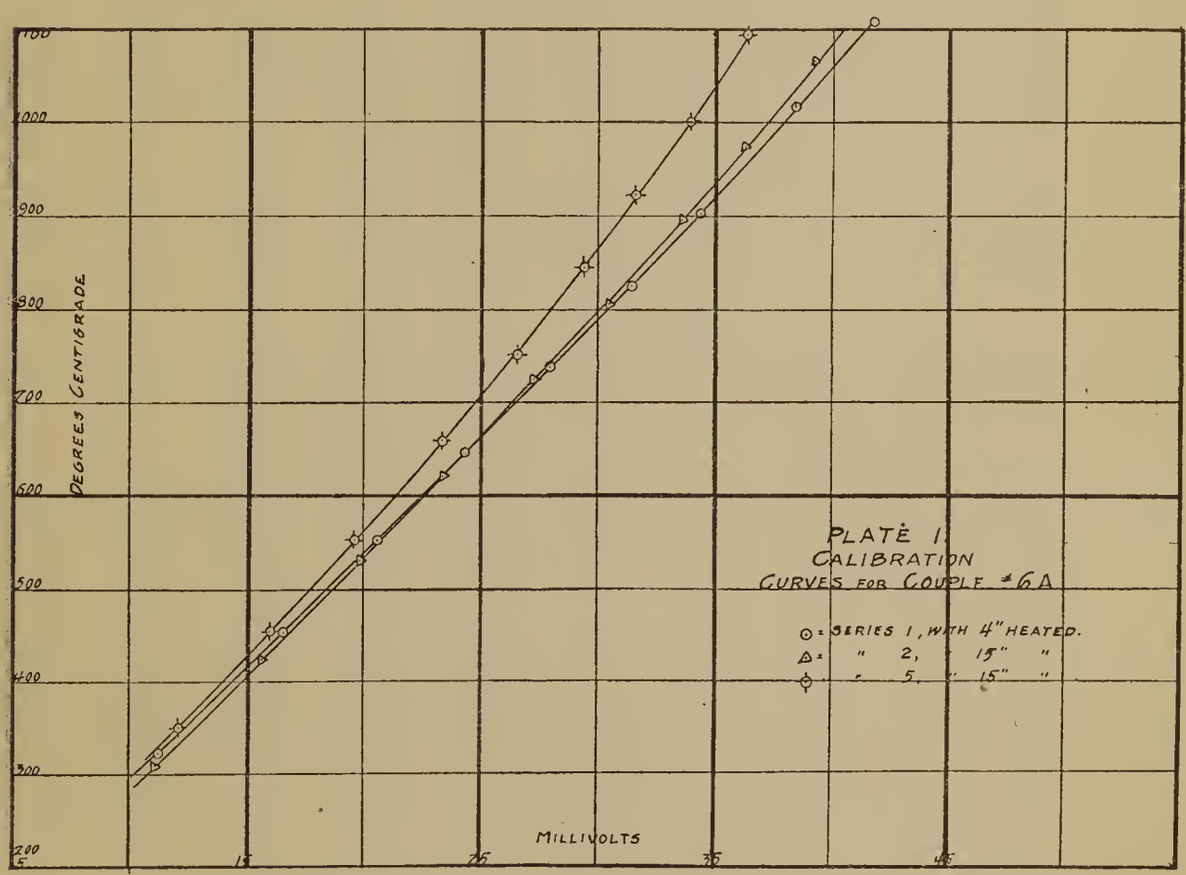

FIG. 1.

Couple No. 20 was made of two No. 16 B. & S. gauge wires, one of which was covered with an asbestos sleeve, and then a coarse asbestos cord was wrapped around both of the wires. No paint covering of any sort was used apparently on this couple. The negative element was a nickel-copper alloy, and the positive element an iron-manganese alloy.

OBSERVATIONS.

The results of the tests on couple No. 6A appear to show that this couple is resistant to oxidation, and was in about as good condition at the end as in the beginning of the series of tests. The asbestos covering appeared to be the most robust and satisfactory of the entire lot. The data of Tables I to V and the curve in Plate 1 show that there is a slight lack of homogeneity in the couple as evidenced by the variation in calibration with

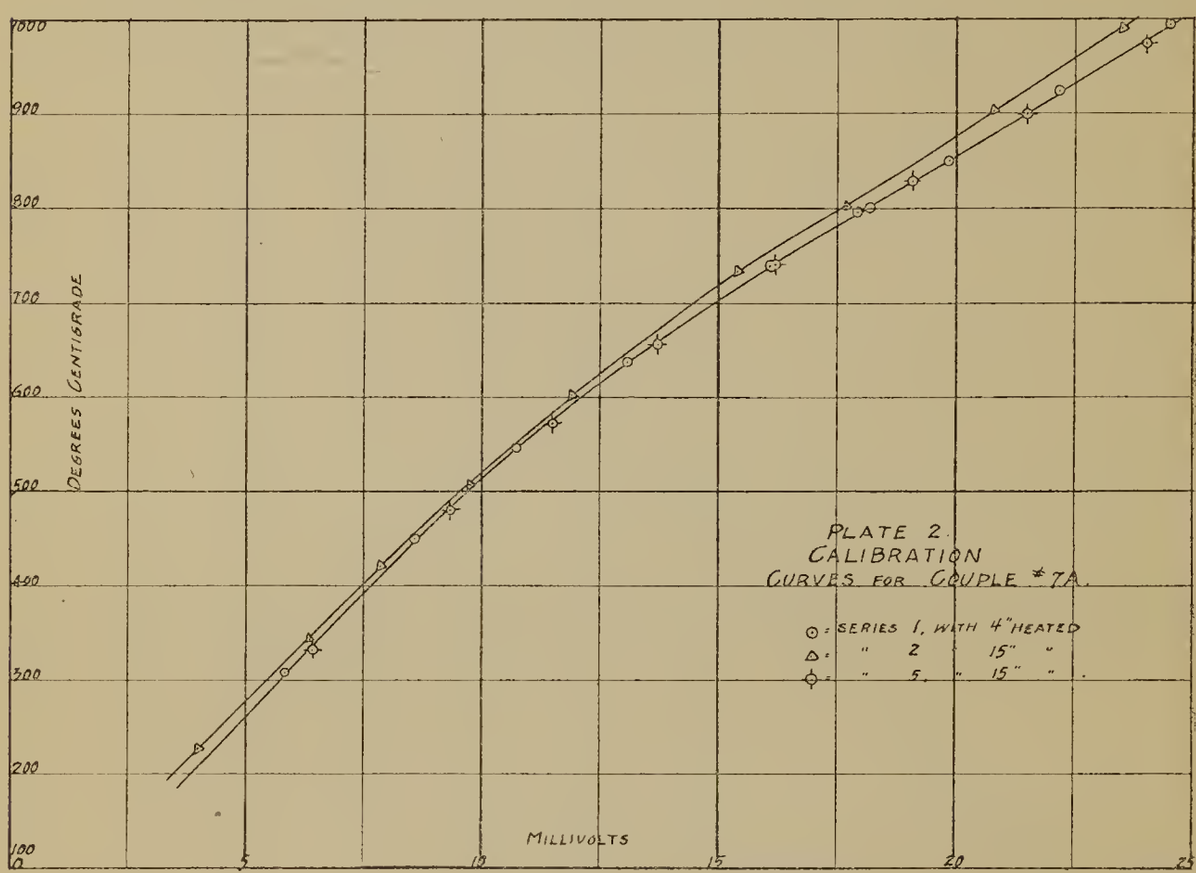

FIG. 2.

4 inches and 15 inches, respectively, of the couple heated, the difference being about 25° at 40 millivolts. The treatments at 400° and 600° C. had little effect upon the calibration of the couple. The treatment at 800° C. had, however, a marked effect upon the calibration, throwing the curve up so that at 35 millivolts the difference between the indications of the initial and final calibrations was nearly 125° C. This large difference is due apparently to some marked change in the structure of one of the wires composing the couple.

The data and curves for couple No. 7A are shown in Tables

I to V and Plate 2. From Plate 2 it is apparent that the calibration curves of this couple are far from being a straight line. The calibrations with 4 inches and 15 inches, respectively, of the couple immersed in the furnace coincide very closely up to 600° C. Above this point the curves separate so that at about 24 millivolts there is a difference of 25 degrees in the indications of the temperature, thus showing the effect of heterogeneity of the wires upon the indication of the couple. It is interesting, however, to note that the heat treatments at 400°, 600° and

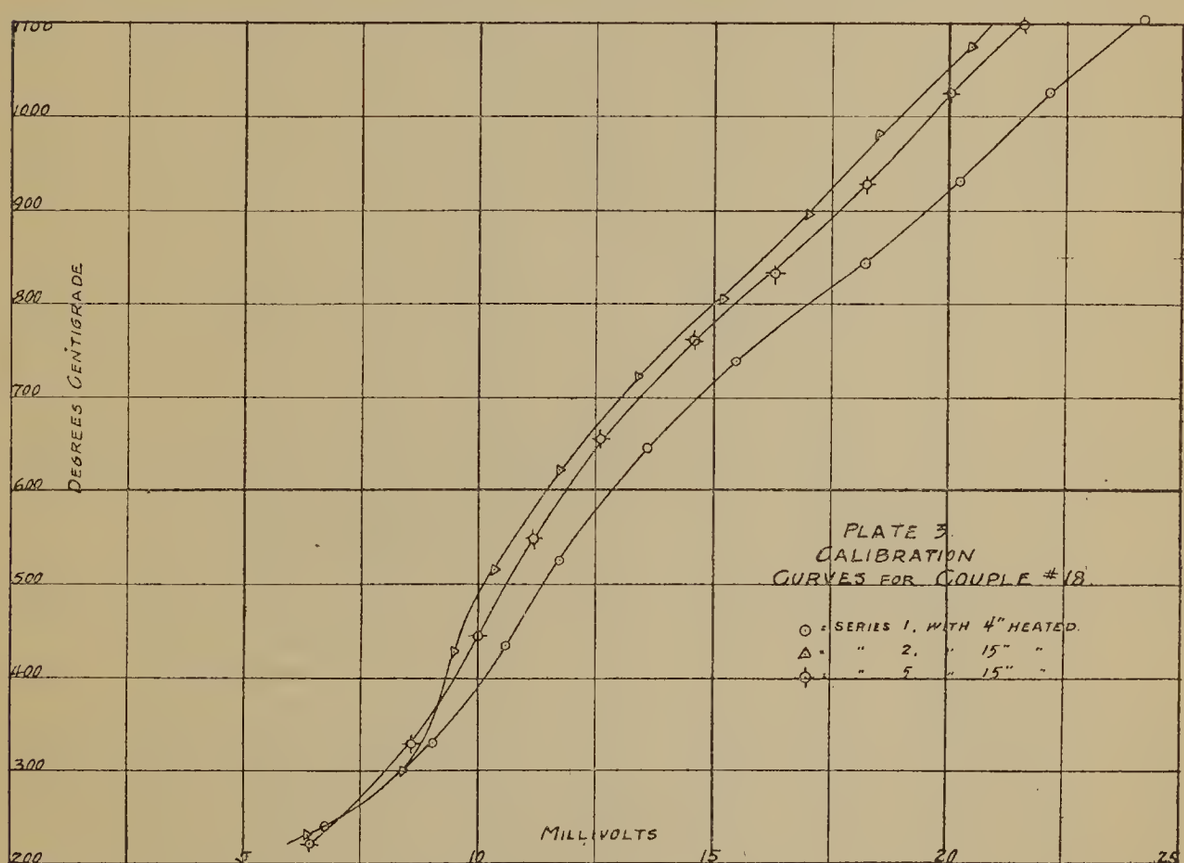

FIG. 3.

800° C., respectively had very little effect upon the constancy with which this couple would indicate the temperature. This couple is robust, and stood the prolonged heat treatments and calibrations without any signs of damage; even the asbestos covering appeared to be in good condition.

Couple No. 18 had a lower electromotive force than either of the two preceding ones. It was found that the iron wire in the couple started to oxidize during the second calibration. As a result of the heat treatment at 400° C. and the third calibration, one of the wires was badly swollen, and after the treatment at

800° it was oxidized so completely that it broke during the subsequent calibration. An examination of the wire revealed the fact that it was oxidized for a distance of about 6 inches beyond the hot junction. It seems likely that the metal of this wire had combined with the silicon of the carborundum paint and formed ferrosilicon. The other wire and its covering were in very good condition with the exception of the carborundum paint, which peeled badly during the first calibration. The data and curves for this couple are shown in Tables I to V and Plate 3.

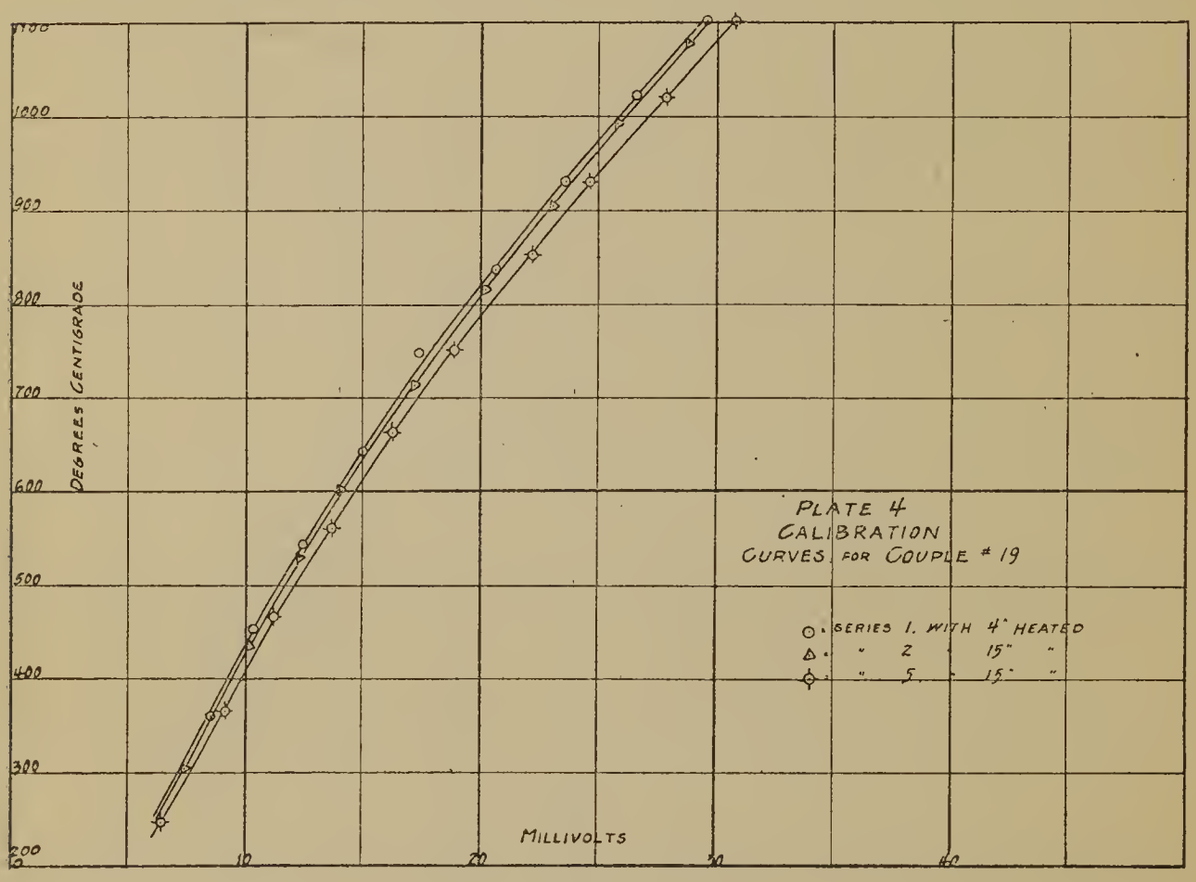

FIG. 4.

Inspection of Plate 3 will show that for an electromotive force of 20 millivolts there is an apparent difference in indication of the temperature by about 130° for a depth of immersion of 4 inches and 15 inches, respectively. This difference is due, no doubt, to a lack of homogeneity of the wires and certain recalescent effects. The calibration curve after the treatment at 400° C. falls between the first and the second calibration curves, showing that some of the heterogeneity has been partially removed. The calibration curves resulting from the treatments at 600° and 800° C. are thrown between the curve after the treatment

at 400° and the first calibration with 15 inches of the couple immersed. It is also apparent that at 9 millivolts there is a point of a marked inflection and that the slope of the curve at this voltage is different for all the curves. Thus it would seem that there would be considerable difficulty in arranging a proper scale for the indicating instrument supplied with this couple.

Couple No. 19 showed very robust qualities, the metal being in fine shape at the end of the series of tests. The asbestos sleeve with which one wire was covered had been completely destroyed.

FIG. 5.

The data and curves in Tables I to V and Plate 4, respectively, show a gradual change in the calibration after each treatment, amounting to about 10 degrees at the higher temperature. The couple appears to be homogeneous, as there is only a slight difference in the calibration curves for a depth of immersion of 4 inches and 15 inches. The curve is very nearly a straight line. The discrepancy between the first and last calibrations is about 40° at 30 millivolts.

The data and curves of couple No. 20 in Tables I to V and Plate 5 show that the variation of millivolts with temperature is

almost linear. This couple gives a higher electromotive force for a given temperature than any of the other couples. It is, however, less resistant to oxidation. This may be due to the fact that the wires composing the couple are only about one-half the diameter of the other couples. After the treatment at 800° C. one of the wires had been oxidized so that it was only about one-half its original size, while the other wire had swollen to about twice its normal size. Both wires had become very brittle and crystalline to within 6 inches of the cold junction. At the end of the last calibration one of the wires broke in two and spoiled the couple. The asbestos covering on this couple remained in fair shape during the entire series of tests. The calibration curves for 4 inches and 15 inches immersion show a slight variation in voltage for a given temperature, indicating that the couple is somewhat heterogeneous. This difference in temperature for a given voltage is less than 20°, which is doing very well for a couple made of this sort of material. The heat treatments at 400°, 600° and 800° C. did not seem to have very much effect upon the change in calibration. This difference is so small that it compares favorably with the changes which frequently take place in high-grade platinum couples.

TABLE I.

Data of Series 1. First Calibration. Couple 4 Inches in Furnace.

Couple No. 6A		Couple No. 7A		Couple No. 18		Couple No. 19		Couple No. 20	
C°	M. V.	C°	M. V.	C°	M. V.	C°	M. V.	C°	M. V.
324	11.39	316	5.86	240	6.75	360	8.41	318	15.70
454	16.64	450	8.59	330	9.09	452	10.40	445	22.51
554	20.73	545	10.76	435	10.66	542	12.45	540	27.66
646	24.41	636	13.10	525	11.75	643	14.98	634	32.99
737	28.03	738	16.12	645	13.64	749	17.34	721	38.11
825	31.51	800	18.27	738	15.49	836	20.65	825	44.85
906	34.36	924	22.21	843	18.20	931	23.60	896	49.41
1018	38.45	993	24.52	933	20.28	1022	26.57	997	55.02
1115	41.43			1026	22.19	1107	29.58		
				1109	24.16				

Table II.

Data of Series 2. Calibration with 15 Inches of Couples Heated.

Couple No. 6A		Couple No. 7A		Couple No. 18		Couple No. 19		Couple No. 20	
C°	M. V.	C°	M. V.	C°	M. V.	C°	M. V.	C°	M. V.
310	11.20	226	3.99	230	6.32	304	7.39	245	11.99
425	15.72	337	6.40	325	8.34	436	10.12	352	17.97
532	19.91	421	7.88	428	9.49	531	12.29	441	22.75
621	23.43	518	9.75	516	10.38	600	14.04	530	27.62
725	27.30	628	12.42	621	11.76	715	17.21	653	34.67
808	30.55	733	15.40	723	13.43	815	20.17	700	37.66
897	33.67	806	17.72	805	15.23	906	23.13	783	42.85
975	36.30	906	20.81	895	17.05	994	25.84	893	49.84
1067	39.19	987	23.47	981	18.51	1078	28.81	984	55.17
				1072	20.46				
				1174	22.77				

Table III.

Data of Series 3. Calibrations After Treatment at 400° C., with 15 Inches Heated.

324	11.81	315	6.02	218	6.33	321	7.88	323	16.55
429	16.05	440	8.28	346	8.79	441	10.36	445	23.10
534	20.42	540	10.49	436	9.81	528	12.36	543	28.52
618	23.56	621	12.47	520	10.87	639	15.25	634	33.87
723	27.26	709	15.22	628	12.27	719	17.56	724	39.11
808	30.45	808	18.47	713	13.86	825	20.59	815	45.19
912	33.97	895	21.34	825	16.43	900	23.10	900	50.66
991	36.63	993	24.81	900	18.20	998	26.37	997	56.54
1090	39.53			990	19.82	1085	29.37		
				1080	21.48				

Table IV.

Data of Series 4. Calibrations After Treatment at 600° C., with 15 Inches Heated.

343	12.71	355	7.08	213	6.04	355	8.94	316	16.38
454	17.09	436	8.68	358	9.32	450	10.83	429	22.30
546	20.76	545	11.04	471	10.32	555	13.33	546	28.83
657	24.80	642	13.41	540	11.05	650	15.80	637	33.83
758	28.66	739	16.17	635	12.21	746	18.31	736	39.77
834	31.36	819	18.64	729	13.82	843	21.64	810	44.75
921	34.33	917	21.69	834	16.14	911	23.85	894	50.16
1006	37.09	995	24.37	912	17.71	1013	27.32	985	55.25
1110	40.29			993	19.12	1096	30.19		
				1102	21.13				

TABLE V.

Data of Series 5. Calibrations After Treatment at 800° C., with 15 Inches Heated.

Couple No. 6A		Couple No, 7A		Couple No. 18		Couple No. 19		Couple No. 20	
C°	M. V.	C°	M. V.	C°	M. V.	C°	M. V.	C°	M. V.
350	12.20	332	6.47	222	6.42	246	6.35	324	16.88
455	16.12	476	9.31	330	8.61	365	9.02	459	24.12
553	19.78	571	11.51	446	9.96	465	11.18	553	29.40
658	23.40	654	13.73	554	11.18	562	13.61	645	34.59
752	26.61	740	16.19	655	12.61	662	16.30	735	40.09
845	29.49	827	19.12	763	14.65	750	18.88	835	46.93
922	31.72	900	21.53	838	16.34	854	22.22	928	52.70
1004	34.03	974	24.07	927	18.25	930	24.71	980	54.77
1095	36.40			1025	20.08	1020	27.95		
				1099	21.58	1102	30.83		

CONCLUSIONS.

This series of investigations seem to point to the fact that it is possible to obtain a base metal couple which is reasonably homogeneous and will give indications of temperature which are sufficiently constant to meet the needs of perhaps the greatest number of requirements for high temperature measurements in the industries.

There are some instances where the temperature is to be known to an accuracy better than 25°, and for such requirements it would be necessary to use a higher grade of equipment, and, furthermore, for such requirements it will be necessary to make frequent calibrations.

It would seem reasonable that the makers of thermocouples determine from the purchaser the particular conditions under which the couple is to be used and that the calibration of this couple be made for the same length of immersion at which the purchaser is going to use it.

A couple which will indicate an apparent difference of temperature amounting to about 130 degrees C. when immersed at different lengths in the bath or furnace is not giving the service that ought to be rendered.

From these tests it appears that not all manufacturers are

careful in sending out couples which have been thoroughly annealed to remove all strains resulting from mechanical treatment. Once the couple has attained its permanent structure, there does not seem to be much change in the electromotive force.

Laboratory of Chemical Engineering,
University of Wisconsin,
Madison, Wis.

A paper presented at the Twenty-fourth General Meeting of the American Electrochemical Society, at Golden, Col., September 11, 1913, President E. F. Roeber in the Chair.

OSMIUM-PLATINUM—A NEW ALLOY.

PRELIMINARY PAPER.

By F. Zimmermann.

Of the several metals of the platinum group, platinum, palladium, iridium and rhodium have been most generally employed in the industrial arts, either alone or in combination as bivalent alloys. Of the latter, iridium-platinum is the best known, but the growing scarcity of iridium has led to the search for other combinations of the metals of this group yielding alloys possessing physical and chemical properties of equal if not greater value. The rarer metals of the platinum group are not easily obtained in great purity, and because of this fact but little success has heretofore been obtained when combining them as bivalent alloys. Furthermore, the strong affinity of osmium for oxygen has increased the difficulty of making alloys of it with other metals in definite proportions. After much experimentation by the author, highly refined platinum and osmium have been successfully combined in widely varying proportions yielding alloys of commercial value. While the two metals may be combined in almost any proportion, alloys containing from 1 to 10 percent of osmium and 99 to 90 percent of platinum are chiefly used.

Great purity of the components is essential, as the presence of small percentages of other elements appears to be very detrimental to the properties of the resulting alloy. According to the chemical and physical behavior, it seems that one part of osmium in an alloy with platinum will take the place of two and one-half times its weight of iridium. The osmium-platinum alloy is very acid-resisting, and for this reason may be of great service in the electrochemical industry. Its electrical resistance is considerably higher than that of an iridium-platinum alloy of the same percentage composition. The alloy further possesses

great hardness and tensile strength. Wires of the finest size are drawn with comparative ease.

A thorough study of the alloy is in progress, and definite results of tests may be of sufficient interest for a later paper.

The alloy has been patented—U. S. Patent No. 1,055,199, of March 4, 1913.

Research Laboratory of Baker & Co., Inc.,
Newark, N. J.

DISCUSSION.

F. A. LIDBURY: Though this is only a preliminary paper, it might have been of interest if a preliminary guess at the price at which this alloy is going to be marketed had been added.

F. ZIMMERMANN (*Communicated*): The price of the osmium-platinum alloy will vary according to market conditions, but, as a basis of calculation, I would state that it will undoubtedly follow the platinum market and the alloy will be priced at approximately the same rate as the iridio-platinum alloys carrying a corresponding percentage of iridium.

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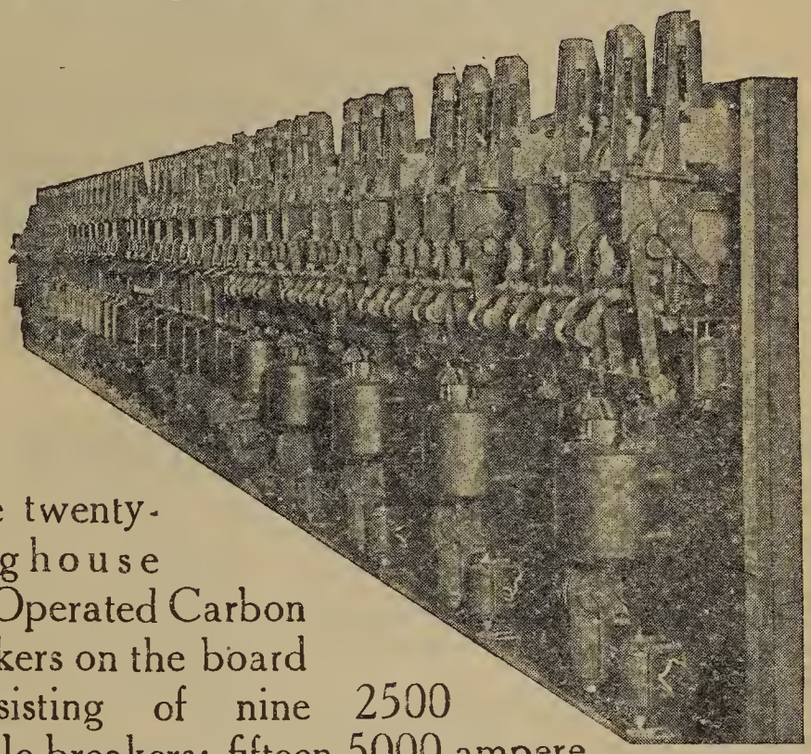

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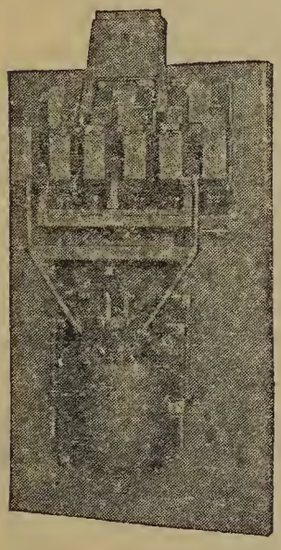

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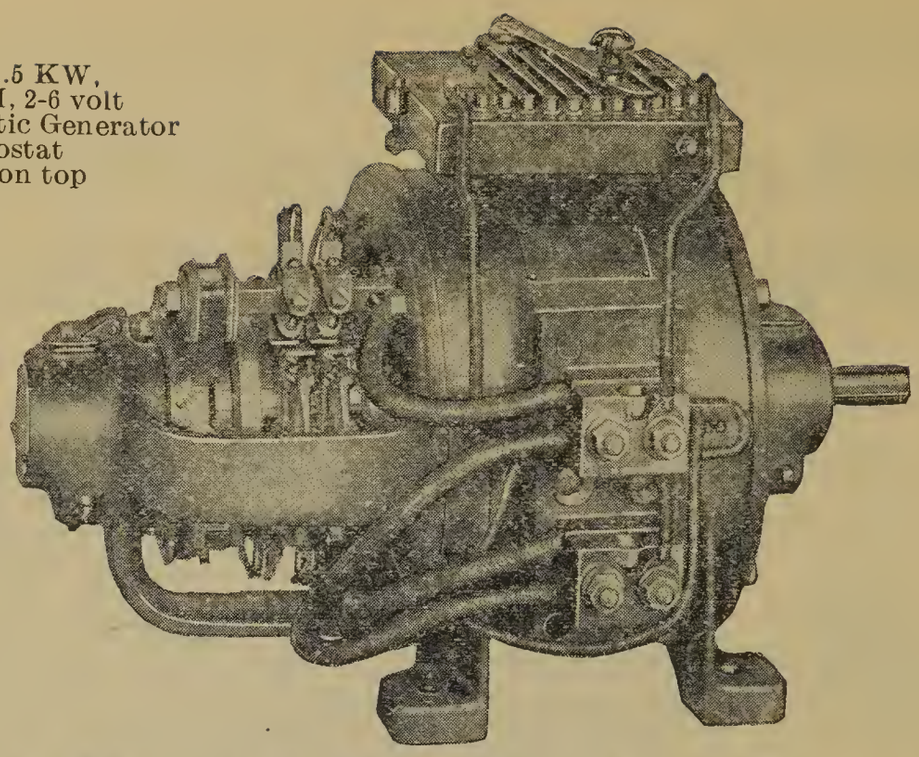

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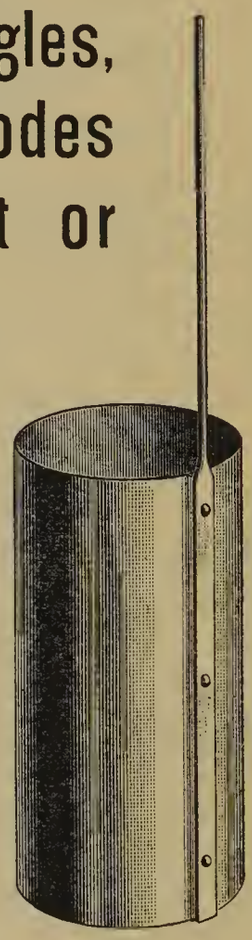

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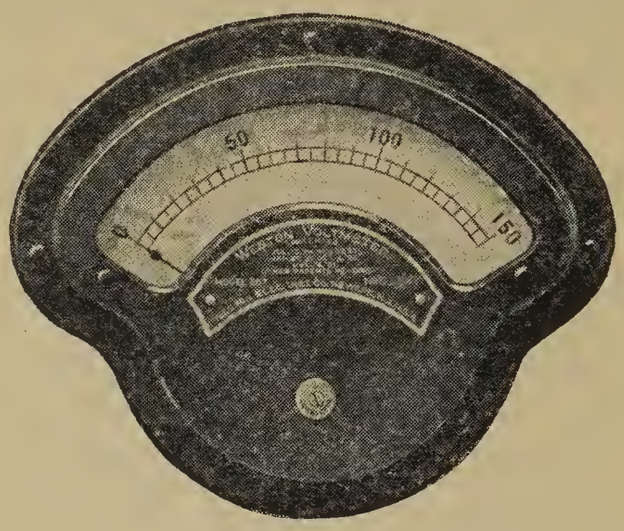

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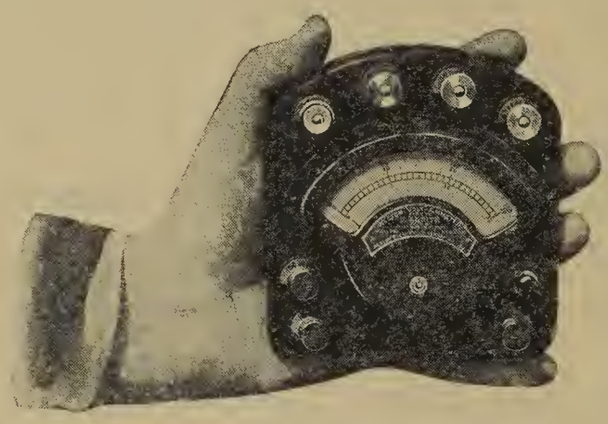

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